

Response of RJ Lee Group to EPA Region 9 (Meer) Letter dated March 9, 2006

Regarding
Evaluation of EPA's Analytical Data
from the
El Dorado Hills Asbestos Evaluation Project

Exhibit C

Date: July 2006

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Preface

In October 2004, the U.S. Environmental Protection Agency (EPA) Region 9 conducted a series of tests in and around El Dorado Hills (EDH), California, to assess the potential exposure of residents to naturally occurring asbestos fibers. EPA released a report of its results to the general public in May 2005 [El Dorado Hills Naturally Occurring Asbestos Multimedia Exposure Assessment El Dorado Hills, California: Preliminary Assessment and Site Inspection Report - Interim Final]. At the request of the National Stone, Sand & Gravel Association (NSSGA), RJ Lee Group, Inc. (RJLG) conducted a review of EPA's May 2005 report and underlying data and issued a report (dated November 2005) entitled "Evaluation of EPA's Analytical Data from the El Dorado Hills Asbestos Evaluation Project". EPA issued a letter dated March 9, 2006 to RJLG and NSSGA requesting the submission of supporting documentation to RJLG's November 2005 report. On April 20, 2006, EPA issued a report entitled "Response to the November 2005 National Stone, Sand & Gravel Association Report Prepared by the R.J. Lee Group Inc [sic] 'Evaluation of EPA's Analytical Data from the El Dorado Hills Asbestos Evaluation Project'" (Region 9 April 20 Response). In addition to the sequence of reports listed above, in an undated letter, Mr. Gregory Meeker, USGS and a consultant to the EPA, prepared a "Response to Questions Submitted by Dr. Vicki Barber, Superintendent of Schools, El Dorado County, California regarding Asbestiform Amphiboles". Dr. Barber's questions were submitted to Dr. Robert (Bob) Virta, USGS, in an email dated February 1, 2006

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Introduction

This document serves as RJ Lee Group's (RJLG) response to the EPA Region 9 (Meer) Letter dated March 9, 2006, requesting information related to RJLG's November 2005 report entitled "Evaluation of EPA's Analytical Data from the El Dorado Hills Asbestos Evaluation Project". RJLG is pleased to be provided the opportunity to clarify any ambiguity in its earlier report and to respond to the comments and concerns expressed by EPA Region 9. RJLG has attempted to provide complete, clear, and concise descriptions of the technical basis of its approach to the issues and the corresponding underlying methodologies.

RJLG has prepared responses to the EPA requests in the same order that they were presented in the March 9th letter. Detailed responses to individual questions are provided in the following sections. Detailed supporting documentation is provided in separate attachments. The attachments are labeled according to the section of the text of this document in which they are mentioned. RJLG will be glad to address any questions regarding our responses.

RJLG believes that the EPA Region 9 April 20 Response, together with Mr. Meeker's Response to Dr. Vicki Barber's questions, raise policy issues that require technical input. To that end RJLG has made commentary in the pages following (see section entitled "Executive Overview and Policy Implications") before addressing the specific questions put forth by EPA Region 9 in the (Meer) March 9 Letter.

This document, beginning at Section 1.0, Response to EPA Region 9 Questions regarding the R.J.Lee [sic] Report (attached to the EPA Region 9 March 9 Letter) includes the specific request from the EPA *shown in italics*, preceded by the phrase [EPA Request] in bold. The RJLG response follows each request.

Executive Overview and Policy Implications

In a letter¹ (hereafter referred to as Meer) dated March 9, 2006, EPA Region 9 requested that RJ Lee Group (RJLG) supplement its commentary on the El Dorado Hills Study. Subsequent to its March 9, 2006 letter, EPA Region 9 published a response to many of the issues raised in the RJLG report. Additionally, Mr. Greg Meeker² of USGS (and on behalf of EPA) provided Dr. Vicki Barber with comments on portions of the RJLG analyses.

RJLG's responses to the major issues and questions raised in the documents mentioned above are summarized here.

EPA's proposed change to asbestos analytical methodology.

The most substantive issue raised in EPA Region 9's response is a proposed modification to the analytical methods for defining and measuring asbestos in a naturally occurring environment. Region 9 proposes that: "amphibole or serpentine minerals that are asbestiform and meet the size definition of phase contrast microscopy (PCM) fibers should be counted as asbestos – regardless of the manner by which they were formed." This quotation, in and of itself, is contradictory – asbestiform fibers are formed over geological time as the minerals develop into fibers; nonasbestos particles are fractured or broken into (sometimes) elongated particles – they are not formed as fibers and are therefore not asbestiform or asbestos.

EPA Region 9's definition implies that any amphibole or serpentine particle longer than 5 μ m with a minimum 3:1 aspect ratio is treated as asbestos. Region 9 further suggests that the risk assessments based on commercial asbestos exposures are suitable for assessing the significance of airborne exposures to amphibole and serpentine particles meeting this definition in a mixed dust "naturally occurring environment," regardless of how the particles were formed.⁴

The geological/mineralogical definition of asbestos is an essential element of the process by which potential asbestos exposure is detected and assessed.

A second argument in Region 9's response is that the geological/mineralogical definition of asbestiform ("bundles of flexible, readily separable fibers, of nearly constant diameter, having parallel sides and a high aspect ratio") is applicable to environments with commercial asbestos exposures but not applicable to environments with exposures to unprocessed naturally occurring asbestos. Commercial asbestos *is* naturally occurring

¹ D. Meer (2006). Letter to W. Ford and to R. Lee, dated March 9, 2006.

² G. Meeker (undated). Response to questions submitted by Dr. Vicki Barber, Superintendent of Schools, El Dorado County, California regarding asbestiform amphiboles. Hereafter referred to as Meeker 2006.

³ EPA Region 9, April 20, 2006. Response to the November 2005 NSSGA Report. Page 11.

⁴ Ibid, page 2.

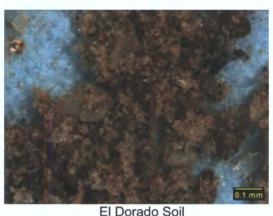
asbestos that has had cleavage fragments and other nonasbestos particles removed during processing.

Naturally occurring asbestos (NOA) is identified through the observation of localized veins of asbestos fibers with the geological/mineralogical characteristics of asbestos. NOA can be found in El Dorado County and other parts of California. Broadening the definition of asbestos would result in increased confusion about the location and extent of asbestos minerals since laboratories may report the presence of asbestos in samples of ordinary rock that contain only nonasbestos amphibole minerals.

Region 9 suggests that naturally occurring asbestos may somehow be different than commercial asbestos. As seen (left, Figure 1), a sample from Harvard Way, El Dorado County, has "classic" characteristics of asbestos. These characteristics are not observed in soil particles from the EPA test area in El Dorado Hills (right, Figure 1).



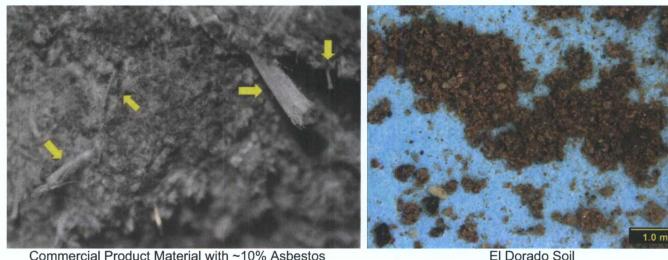
Asbestiform Amphibole - Harvard Way 0.6 mm Field of View



0.6 mm Field of View

Figure 1. Asbestiform amphibole from Harvard Way collected within several hundred yards of the EPA El Dorado Hills test area (left) as observed under low magnification optical microscopy. The soil sample from an EPA El Dorado Hills test area (right), does not exhibit any asbestos characteristics.

Figure 2 shows a bulk sample of commercial product material that was determined to contain 5% to 10% asbestos. At this percentage, the asbestiform component is clearly visible. Asbestos TEM Laboratories reported that the soils of El Dorado Hills contained 5% to 10 % asbestos, but as seen in Figure 2, the soil does not contain any evidence of asbestiform components.



Commercial Product Material with ~10% Asbestos 6.0 mm Field of View

6.0 mm Field of View

Figure 2. Image of commercial product material containing ~10% asbestos (left) compared to the El Dorado soil (right). Yellow arrows point to asbestos structures.

Asbestiform and nonasbestos amphiboles have distinctly different characteristics.

The characteristics of asbestiform particles in NOA specimens are the same as asbestiform particles in commercial asbestos samples. In both cases, the physical characteristics of asbestiform fibers are significantly different than those of nonasbestos fragments. Over the last 40 years a significant body of literature has accumulated demonstrating that potency is most associated with populations of "long-thin" fibers.^{5,6,7,8,9} Populations of nonasbestos fragments have different characteristics than asbestiform populations (Figure 3).

The tremolite asbestos fibers in Figure 3a, produced by shaking an asbestiform bundle from a Jamestown, California sample, have very little variation in diameter and extremely high aspect ratios. Note the bundles and thin curved fibers. In contrast, the nonasbestos

⁵ M. F. Stanton, M. Laynard, A. Tegeris, E. Miller, M. May, E. Morgan, and A. Smith (1981). "Relation of Particle Dimension to Carcinogenicity in Amphibole Asbestoses and Other Fibrous Minerals", J of the National Cancer Institute, <u>67</u>, p. 965-975.

⁶ A. Wylie, K. F. Bailey, J. Kelse, R. Lee (1993). "The Importance of Width in Fiber Carcinogenicity and Its Implications for Public Policy", AIHA Journal, <u>54</u>, p. 239-252.

⁷ J. M. G. Davis, J. Addison, C. McIntosh, B. G. Miller, and K. Niven (1991). "Variations in Carcinogenicity of Tremolite Dust Samples of Differing Morphology", Annals New York Academy of Sciences, <u>643</u>, p. 473-489.

⁸ D. W. Berman and K. Crump (2003). Technical support document for a protocol to assess asbestos-related risk," EPA, U.S. Environmental Protection Agency, Revision of original from September 4, 2001, Peer-reviewed consultation held in San Francisco on February 25-26, 2003.

⁹ E. D. Kuempel, L. T. Stayner, J. D. Dement, S. J. Gilbert, and M. J. Hein (2006). "Fiber Size-Specific Exposure Estimates and Updated Mortality Analysis of Chrysotile Asbestos Textile Workers", presented at the Society of Toxicology meeting, March 6, 2006.

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tremolite particles in Figure 3b have the characteristics described by Campbell et al¹⁰ of nonasbestos amphiboles -- blocky or angular ends with no evidence of internal structure, and having visible crystal faces, non-parallel or stepped sides, and a non-fibrous appearance. It is apparent, even to the untrained eye, that the Jamestown sample has the characteristics of a sample containing asbestiform particles as defined in the NIST SRM 1867a certificate of analysis,¹¹ and the nonasbestos tremolite has the characteristics of the nonasbestos populations defined by Campbell et al.¹⁰

¹⁰ Campbell, W. J., R. L. Blake, L. L. Brown, E. E. Cather, J. J. Sjoberg (1977), 'Selected Silicate Minerals and Their Asbestiform Varieties - Mineralogical Definitions and Identification-Characterization', Bureau of Mines, United States Department of Interior, Information Circular 8751, pp. 1-55.

¹¹ National Institute of Standards and Technology, 2003. Certificate of Analysis; Standard Reference Material® 1867a, Uncommon Commercial Asbestos. Available:

 $[\]frac{https://srmors.nist.gov/certificates/1867a.pdf?CFID=4653224\&CFTOKEN=3b1dee8a6980cca6-F6F7B807-B6AE-6927-254940B41F0F309C\&jsessionid=b4302e2788e7\$DB\$FE\$A.\ Accessed May, 2006.$

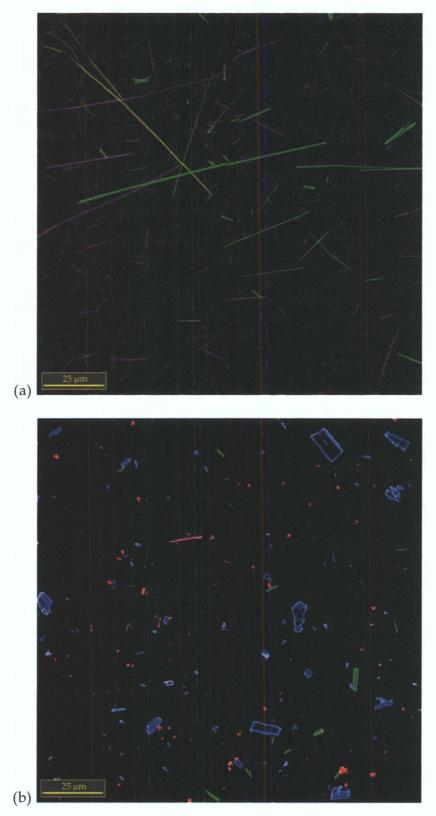


Figure 3. Tremolite asbestos from Jamestown, California sample (a) compared to a nonasbestos tremolite (b) showing distinct morphological differences.

When particles from the El Dorado Hills Study are compared to asbestiform and nonasbestiform populations, the El Dorado samples show widths that are significantly wider than those from Jamestown asbestiform tremolite and Shinness nonasbestiform tremolite. Jamestown asbestiform tremolite was found to produce 100% tumors in the Addison-Davis experiments. Shinness nonasbestiform tremolite was not shown to cause asbestos-like disease in the Addison-Davis experiments.⁷ Nonasbestos populations have few thin particles when compared to asbestos populations, including those found in the El Dorado Study (Figure 4).

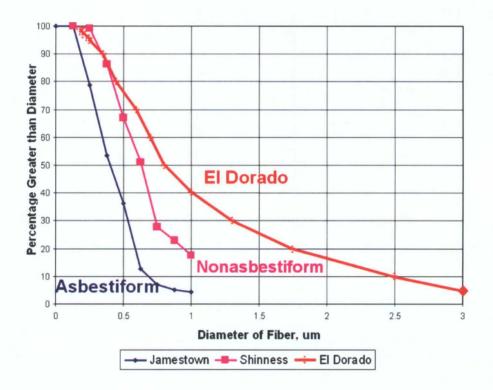


Figure 4. Comparison of particle size populations between asbestiform amphibole from Jamestown, nonasbestiform amphibole from Shinness, and amphibole particles from El Dorado Hills soil samples.

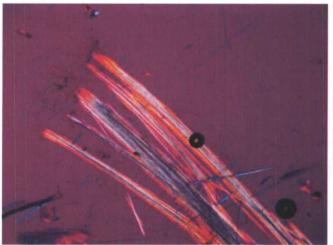
Current analytical procedures use the geological definition of asbestos to distinguish asbestos fibers from nonasbestos amphiboles and serpentines.

EPA suggests in their April 20th response that it is nearly impossible to distinguish between asbestiform and nonasbestos amphiboles microscopically.

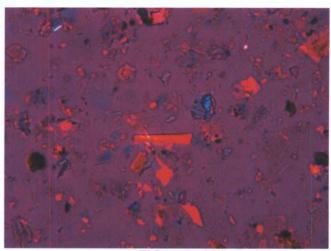
In fact all asbestos analyses depend on the microscopic properties of asbestos fibers as viewed microscopically. Asbestiform is defined in numerous PLM methods as well as in the NIST 1867a SRM certificate of analysis.¹¹ The definition of asbestiform in the NIST SRM 1867a certificate is:

"Asbestiform: crystallizes with the habit of asbestos. These asbestos minerals possess properties such as long fiber length and high tensile strength. Under the light microscope, some portion of these samples exhibit the asbestiform habit as defined by several of the following characteristics: 1) mean aspect ratios ranging from 20:1 to 100:1 or higher for fibers longer than 5 μ m, 2) very thin fibrils, usually less than 0.5 μ m in width, 3) parallel fibers occurring in bundles, 4) fiber bundles displaying splayed ends, 5) fibers in the form of thin needles, 6) matted masses of individual fibers, and 7) fibers showing curvature."

Asbestos counting methods published over the past 30 years define asbestos as the mineral fibers in the asbestiform habit of six specified mineral species. Asbestiform fibers, regardless of their regulatory status, have unique shapes, crystallinity, and surface texture as well as optical properties that differentiate them from nonasbestos particles^{12,13,14,15} (Figure 5). In the PLM, asbestos fibers are characterized by parallel sides, curvature with sufficient length, and proper termination of the ends. In contrast, prismatic and acicular particles have blunt, angular, or stepped ends, parallel and non-parallel sides, recognizable crystal or cleavage faces and an absence of internal fibrosity.



San Andreas Asbestiform Tremolite 40x magnification, 2.5 mm Field of View



El Dorado Soil 40x magnification, 2.5 mm Field of View

Figure 5. Comparison of Asbestiform Amphibole from San Andreas California (left) to the Amphibole Particles observed in El Dorado Hills Soil (right).

¹² W. J. Campbell, R. L. Blake, L. L. Brown, E. E. Cather, and J. J. Sjoberg (1977). "Selected Silicate Minerals and Their Asbestiform Varieties: Mineralogical Definitions and Identification-Characterization", US Bureau of Mines Information Circular, IC 8751.

¹³ A. G. Wylie, R. L. Virta, and E. Sussek (1985). "Characterizing and Discriminating Airborne Amphibole Cleavage Fragments and Amosite Fibers: Implications for the NIOSH Method", AIHA Journal, <u>46</u>, p. 197-201.

¹⁴ Michael E. Beard letter dated 11/03/92 to Sally A. Sasnett of the USEPA (United States Environmental Protection Agency) regarding: Definitions used to define Asbestos Fibers / Asbestos Cleavage Fragments / Aspect Ratios.

¹⁵ R. J. Lee and R. M. Fisher (1979). "Identification of Fibrous and Nonfibrous Amphiboles in the Electron Microscope", Health Hazards of Asbestos Exposure, Annals of New York Academy of Science, <u>330</u>, p. 645-660.

The attributes of asbestos as observed by PLM can also be observed in the electron microscope. It is widely recognized that asbestiform particles have unique shapes and lie in preferred directions in the optical and electron microscopes. The preferred orientation and internal defects common in the asbestos habits give rise to electron diffraction patterns with rows of closely spaced spots. Virtually all methods cite the 0.53 nm row spacing as a characteristic of asbestiform amphiboles. This, coupled with the parallel sides of asbestos fibers, makes asbestos different than the vast majority of cleavage fragments (Figure 6).



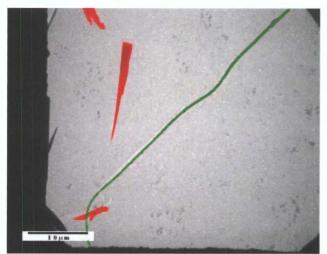
Figure 6. Photographs from EPA Fiber Atlas¹⁶ showing particle image and SAED pattern of asbestos fiber with 0.53 nm spacing defined by Yamate ¹⁷ as characteristic of amphibole asbestos.

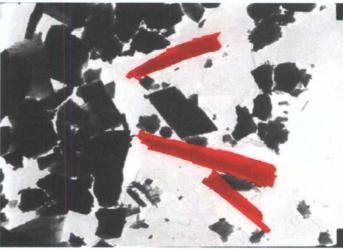
In contrast, when the laboratory does not maintain the requirement for parallel sides, or examine the particles for the recognized physical characteristics of asbestiform particles, serious errors occur. In the analysis performed relative to Figure 7 (right photo), the laboratory performing the analysis made the same error as Lab/Cor in this case, and did not follow the method requirement that fibers are to have "parallel sides" to be countable in the TEM. Instead they counted all particles with a minimum 3:1 aspect ratio as an asbestos fiber, thereby giving rise to a high apparent asbestos concentration that they recognized was questionable but that they did not know how to address.

¹⁶ P. K. Mueller, A. E. Alcocer, R. L. Stanley, and G. R. Smith (1975). "Asbestos Fiber Atlas." U. S. Environmental Protection Agency, EPA-650/2-75-036.

¹⁷ G. Yamate, S. C. Agarwal, R. D. Gibbons (1984). "Methodology for the Measurement of Airborne Asbestos by Electron Microscopy", IIT Research Institute, Contract No. 68-02-3266, July 1984.

In the case of many particles, the distinction between asbestiform fibers and nonasbestos cleavage fragments is obvious as shown in Figure 7.





Harvard Way Asbestiform Amphibole

Taconite Mine Particles

Figure 7. Harvard Way asbestiform amphibole (left-asbestiform fiber highlighted green) compared to amphibole particles recently counted as asbestos fibers from a Taconite mine¹⁸ (Sample 962215; right-counted particles highlighted red) where the laboratory failed to follow counting rules. Note the lack of parallel sides in the particles on the right. These particles should not be counted as asbestos fibers.

When a TEM analyst utilizes the characteristics as defined by the NIST SRM 1867a certificate of analysis, ¹¹ OSHA ID 191¹⁹, and the TEM analytical methods²⁰ to conduct the analysis, the vast majority of nonasbestos particles are excluded from the count of asbestos fibers.

PLM and TEM methods recognize the physical differences between asbestiform and nonasbestos particles.

¹⁸ Haney, Robert A. (2005). U.S. Department of Labor, Mine Safety and Health Administration, Memorandum for Steven M. Richetta, District Manager, Metal and Nonmetal Mine Safety and Health, North Central District, Duluth, Minnesota, "Respirable Dust and Mineral Fiber Investigation at Northshore Mining Company (Mine I.D. No. 21 00831), Silver Bay, Lake County, Minnesota, September 20, 2005.

¹⁹ D. T. Crane (1992). "Polarized Light Microscopy of Asbestos," OSHA Analytical Methods Manual, Method ID-191.

²⁰ U.S. Environmental Protection Agency (USEPA) (1987). Asbestos Hazard Emergency Response Act, 40 CFR Part 763, Appendix A to Subpart E. USEPA, Washington, DC.

Inspection of mines and quarries for the occurrence of asbestos is performed as a means of ensuring that products will not have harmful levels of asbestos. Field geologists and mining engineers rely on the geological and mineralogical definition of asbestos in performing inspections for the presence of asbestos and potential for exposure to asbestos. Current regulations and analytical methods measure the concentration of the asbestos minerals. Analytical methods, including OSHA 191 and EPA 600/R-93/116, define asbestos in terms of its geological/mineralogical characteristics (aspect ratio, flexibility, bundles). Yamate⁷⁰ and the EPA Asbestos Fiber Atlas²¹ use microscopic characteristics of asbestiform fibers to define the attributes of asbestos.

OSHA Method 191 states:

"Most cleavage fragments of the asbestos minerals are easily distinguishable from true asbestos fibers. This is because true cleavage fragments usually have larger diameters than 1 μ m. Internal structure of particles larger than this usually shows them to have no internal fibrillar structure. In addition, cleavage fragments of the monoclinic amphiboles show inclined extinction under crossed polars with no compensator. Asbestos fibers usually show extinction at zero degrees or ambiguous extinction if any at all. Morphologically, the larger cleavage fragments are obvious by their blunt or stepped ends showing prismatic habit. Also, they tend to be acicular rather than filiform.

. . . Use the same morphological clues for electron microscopy as are used for light microscopy, e.g. fibril splitting, internal longitudinal striation, fraying, curvature, etc.

...When particles are below the limit of the PLM, OSHA recommends using the SEM or TEM for thin fibers, i.e. those less than 1 micrometer in width. "Where the particles are less than 1 μm in diameter and have an aspect ratio greater than or equal to 3:1, it is recommended that the sample be analyzed by SEM or TEM if there is any question whether the fibers are cleavage fragments or asbestiform particles." 22

Campbell¹⁰ describes the characteristics of cleavage fragments and provides drawings of representative shapes. These are used in the optical microscope or scanning electron microscope (SEM) to resolve questions about the morphological distinctions.

Thus the combinations of these defining characteristics are routinely used in the (PLM), SEM, or (TEM) methods to distinguish asbestiform from nonasbestiform amphiboles with a high degree of reliability. Where there is uncertainty, combinations of techniques to

²² OSHA ID-191, Section 3.5: Analytical Procedure.

²¹ P. K. Mueller, A. E. Alcocer, R. L. Stanley, and G. R. Smith (1975). "Asbestos Fiber Atlas." U. S. Environmental Protection Agency, EPA-650/2-75-036.

examine particles in more than one microscope can be used to improve the discrimination.²²

Many laboratories, whose principal business is analyzing samples taken after asbestos abatement projects, do not have the knowledge of the literature or the training to make the necessary distinctions. They need supplemental guidance and training to effectively analyze samples from mixed mineral environments.²³

The amphibole particles in the El Dorado Hills Study do not have the characteristics of asbestiform particles.

Asbestos fibers have a nearly constant diameter. For amphiboles, this diameter is generally less than 0.3 μm , but they have been reported to have diameters up to 0.5 μm . Long asbestiform fibers have about the same width as short asbestiform fibers, thus the length is independent of the width. In contrast, long cleavage fragments tend to be thicker in diameter. The populations of particles in the El Dorado data exhibit a direct proportionality between the lengths and widths of the fibers (Figure 8). The average aspect ratio is 6.3:1 for elongate particles longer than 5 μm . Only a portion of these particles that are three times longer than they are wide have parallel sides. The amphibole particles in the soil have a high index of refraction and maximum extinction angles uniformly above 10 degrees which is characteristic of hornblende, not actinolite or tremolite. These particles exhibit the characteristics of a population of cleavage fragments, not asbestiform fibers.

²³ E. Chatfield (2002). Testimony before MSHA, Charlottesville, VA, June 20, 2002, p 144 - 145.

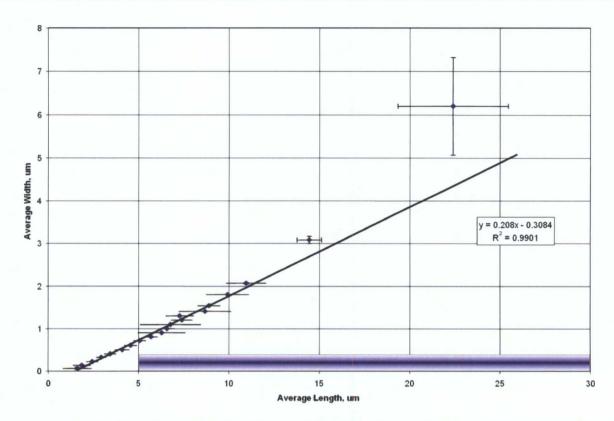


Figure 8. Relationship of average lengths and widths of amphibole particles observed in El Dorado Hills. The Berman-Crump Risk Fiber region (width $< 0.4 \mu m$, length $> 5 \mu m$ is highlighted in blue).

Integrated Risk Assessment Models are based on exposure to commercial asbestos.

Epidemiology studies and animal experiments used for the IRIS and OSHA risk assessment have documented the link between asbestos fibers and disease (asbestosis, lung cancer, and mesothelioma). All of these studies have documented this link on the basis of an exposure to commercially-produced asbestos fibers that were processed and sold as a pure mineral product with only nominal contamination by nonasbestos fragments.

EPA has conducted a review to establish an integrated risk assessment model, Integrated Risk Information System, (IRIS).²⁴ The epidemiology studies referenced in IRIS documents are based on exposures to commercial asbestos fibers, not to mixed atmospheres of asbestos and nonasbestos particles as claimed by EPA Region 9.²⁵ The only epidemiology studies at that time that may have included a substantial population of nonasbestos mineral fibers were those of the chrysotile miners and millers, a study that was explicitly

²⁵ EPA (2006), page 2.

²⁴ US Environmental Protection Agency, Integrated Risk Information System (IRIS), http://www.epa.gov/iris/subst/0371.htm.

excluded from the IRIS model. Berman has noted that extension of a risk model to new environments is dependent on the ability to measure the same type of asbestos fiber population assessed in establishing the risk in the reference studies.^{8,26}

Nonasbestos amphiboles and serpentines have not been associated with asbestos disease.

OSHA thoroughly examined the cleavage fragment issue from 1986 to 1992²⁷ and concluded that cleavage fragments do not present an asbestos risk. Hard-rock mines and quarries often contain nonasbestos amphiboles including hornblende, actinolite, and tremolite, such as that found in the El Dorado Hills Study, but there has been no asbestos-related disease in the mine workers.^{28,29,30} The NSSGA has provided the El Dorado County School District and the EPA with a number of recent literature reviews³¹ conducted since 1992 that support this conclusion.

EPA Region 9's definition of asbestos would have significant economic impact and analytical implications.

The Region 9 definition of asbestos would represent a significant shift in the policy of EPA and would put the policy of the Agency in conflict with OSHA and other Federal Agencies and international organizations.^{32,33,34,35} Given the widespread occurrence of nonasbestos

²⁶ D. W. Berman (2006). "Evaluation of the Approach Recently Proposed for Assessing Asbestos-Related Risk in El Dorado County, California", page 14.

²⁷ OSHA 1992 preamble, found at http://www.osha.gov/pls/oshaweb/owasrch.search_form?p_doc_type=PREAMBLES&p_toc_level=1&p_keyvalue=Asbestos~(1992~-~Original)

²⁸ Y. Honda, C. Beall, E. Delzell, K. Oestenstad, I. Brill, and R. Matthews (2002). "Mortality Among Workers at a Talc Mining and Milling Facility", Ann. Occup. Hyg., <u>46</u>, p. 575-585.

²⁹ K. Steenland and D. Brown (1995). "Mortality Study of Gold Miners Exposed to Silica and Nonasbestiform Amphibole Minerals: An Update with 14 more years of Follow-Up", American Journal of Industrial Medicine, <u>27</u>, p. 217-229.

³⁰ W. C. Cooper, O. Wong, L.S. Trent, and F. Harris (1992). "An Updated Study of Taconite Miners and Millers Exposed to Silica and Non-Asbestiform Amphiboles." JOM, <u>34</u>, p. 1173-1180.

³¹ J. F. Gamble and G. W. Gibbs (2006). An Evaluation of the Risks of Lung Cancer and Mesothelioma from Exposure to Amphibole Cleavage Fragments, Peer Reviewed, Revised and Accepted for Publication in Proceedings of the International Symposium on the Health Hazard Evaluation of Fibrous Particles Associated with Taconite and the Adjacent Duluth Complex St Paul, Minnesota, March 30 – April 1, 2003.

J. Addison and E. E. McConnell (2006). "A Review of Carcinogenicity Studies of Asbestos and Non-Asbestos Tremolite and Other Amphiboles", Peer Reviewed, Revised and Accepted for Publication in Proceedings of the International Symposium on the Health Hazard Evaluation of Fibrous Particles Associated with Taconite and the Adjacent Duluth Complex St Paul, Minnesota, March 30 – April 1, 2003.

B. T. Mossmann (2006). "Assessment of the Pathogenic Potential of Asbestiform vs. Nonasbestiform Particulates (Cleavage Fragments) in *In Vitro* (Cell or Organ Culture) Models and Bioassays", Peer Reviewed, Revised and Accepted for Publication in Proceedings of the International Symposium on the Health Hazard Evaluation of Fibrous Particles Associated with Taconite and the Adjacent Duluth Complex St Paul, Minnesota, March 30 – April 1, 2003.

^{32 1992} OSHA Ruling, preamble

amphiboles and serpentines throughout the US (Figure 9), the adoption of the Region 9 definition on a national basis could have significant economic consequences for both public and private organizations, including school districts in newly developing areas such as El Dorado.³⁶ Commercial entities including real estate developers, the crushed stone, mining, and construction industries would have to commit additional resources to monitoring product quality and potential exposures using sophisticated methods for no measurable health benefit. The long term use of recycled stone products would also be seriously affected by such rulings since there are literally billions of tons of potentially newly re-defined (by Region 9) asbestos-containing stone currently in structures in the US. By classifying nonasbestos particles as asbestos, exported products may be considered "asbestos-containing" in other countries³⁷ thus imposing control measures that will do nothing to improve public health.

³³ D. T. Crane (1992). "Polarized Light Microscopy of Asbestos," OSHA Analytical Methods Manual, Method ID-191.

³⁴ Michael E. Beard letter dated 11/03/92 to Sally A. Sasnett of the USEPA (United States Environmental Protection Agency) regarding: Definitions used to define Asbestos Fibers/Asbestos Cleavage Fragments/Aspect Ratios.

³⁵ Consumer Product Saftety Commision (2000). "CPSC releases Test Result on Crayon." Press Release no. 00-12313. June, 2000. http://www.cpsc.gov/scpscpub/prerel/prhtml00/00123.html. and Langer, A. M., R. P. Nolan. "Mineralogical Analysis of Two "Play Sands" for Their Asbestos Contents." Report to the Consumer Product Safety Commission. November 1986.

³⁶ V. L. Barber letter to Stephen L. Johnson, March 10, 2006.

³⁷ H. Benjelloun (2000). "The European Union's Ban on Asbestos", The Synergist, August 2000, p. 20 – 25.

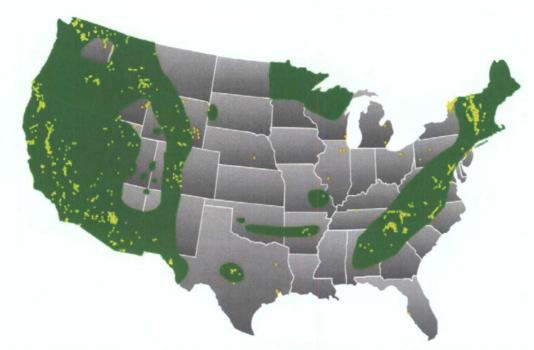


Figure 9. Green shaded areas demonstrate regions where amphibole minerals may likely occur. Yellow dots represent possible locations of where asbestiform minerals may occur according to the USGS database³⁸ (after EPA³⁹).

Improved methodology for evaluating the asbestos content of mixed-mineral dust. ASTM has recognized and addressed the complexity of analyzing asbestos in mixed dust

ASTM has recognized and addressed the complexity of analyzing asbestos in mixed dust atmospheres by developing a rapid screening optical protocol that preserves the information obtained in the conventional PCM analysis but added a discriminate analysis component to identify samples with significant numbers of long, thin fibers (longer than $10~\mu m$ and less than $1~\mu m$ wide).⁴⁰ It is expected that the ASTM method will be published this summer.

If an elevated content of long thin fibers is detected optically, the ASTM method recommends supplemental electron microscope analysis. Currently, the tendency in the USA is to proceed directly from optical to TEM analysis with the belief that the SEM lacks the visibility necessary to image fine fibers. The advent of high resolution digital SEMs has created the possibility for rapid, low-cost screening analysis. These microscopes now

³⁸ E. J. McFaul, G. T. Mason, Jr., W. B. Ferguson, and B. R. Lipin (2000). U.S. Geological Survey Mineral Databases – MRDS and MAS/MILS, U.S. Geological Survey Digital Data Series DDS-52, U.S. Geological Survey, Washington, DC.

³⁹ R. J. Kuryvial, R. A. Wood, and R. E. Barrett (1974). "Identification and Assessment of Asbestos Emissions from Incidental Sources of Asbestos", US EPA 650/2-74-087.

⁴⁰ ASTM (2006). "Standard Test Method for Sampling and Counting Airborne Fibers, Including Asbestos Fibers, in Mines and Quarries by Phase Contrast Microscopy", ASTM D 7200.

Response of RJ Lee Group to EPA Region 9 (Meer) Letter dated March 9, 2006

have the resolution to image the finest chrysotile fibers, yet can scan much larger areas than is feasible with the TEM.

The ASTM optical procedure should be repeated in the SEM, as a supplemental analysis, separating the longer than 10 μ m particles and less than 1 μ m in diameter into those less than 0.4 μ m in width, and those wider than 0.4 μ m. The SEM images can also be used to determine if the particle is asbestiform or nonasbestiform in many instances. The ratio of fine/coarse fibers and estimate of the asbestiform fraction would provide a reliable estimate of the asbestos content of samples from mixed mineral environments.

The current TEM methods should be modernized or amended to provide a clear and unambiguous method for discriminating between asbestos and nonasbestos amphiboles.

Response to EPA "Questions regarding the R.J. Lee Report [sic]" attached to the EPA Region 9 (Meer) Letter dated March 9, 2006

The following sections are written in response to the questions (also referred to as NSSGA and R.J. Lee Group [sic] Information Request). Sections 1 through 13 generally follow the order as presented in pages 3 – 6 of the EPA Region 9 (Meer) March 9 letter.

The EPA request is shown in italics at the beginning of each section and is followed by the RJLG Response.

1.0 EPA Air Samples

[EPA Request] Please list all the analytical techniques (including full method name and reference number) that the R. J. Lee Group [sic] used to evaluate the EPA air samples. Please provide all documents generated as a result of that analysis including: laboratory count sheets, laboratory notes and logbook pages, sketches, images, spectra, diffraction patterns, chain-of-custody forms and other sample tracking sheets. Provide the same information for any and all quality control ("QC") samples analyzed along with the investigative samples and all required calibrations and other technical notes generated during the review of the EPA air samples. Please include a general description of the instruments (including make and model) used in the analyses and provide the analyst's names and qualifications to perform analysis for each of the analytical/preparation methods employed. If no actual laboratory analysis was performed, or if, in addition to laboratory analyses, other reviews were performed, identify such reviews, explain the steps taken for conducting the review, and provide all available documents of such reviews.

1.1 Evaluation of EPA Analyses

RJLG has requested air filters from EPA Region 9. To date, no air filters have been received. As a result, RJLG has performed no analysis of air filters from El Dorado Hills. RJLG will analyze and provide detailed results on any filters Region 9 is willing to provide.

1.2 Evaluation of EPA Data

[EPA Request] If no actual laboratory analysis was performed, or if, in addition to laboratory analyses, other reviews were performed, identify such reviews, explain the steps taken for conducting the review, and provide all available documents of such reviews.

RJLG's review of EPA's air sample data was conducted over a six-month period of time and included an informal, preliminary presentation of the data evaluation at the 2005 ASTM Johnson Conference (see Attachment C-1.2). RJLG's initial review was performed on data submitted by the EPA to the El Dorado Hills (EDH) School District and community members. Although RJLG had requested a complete set of data and documents from the EPA, only a partial set was sent on August 8, 2005, by Karen Ladd

(Ecology and Environment, Inc.). The data were received on two compact discs: "Raw Data Summaries Grouped by Activity" and "Raw Data Summaries Plus Detail Grouped by Activity." The information contained on the "Detail" CD was the same information that was previously provided to RJLG by community members. The information contained on the CDs was limited to a summary page for each sample analysis and the associated count sheets for the sample.

On September 6, 2005, RJLG received a more complete set of data on two CDs. The data on each CD were organized by Lab/Cor job number. Within each job number was the previously provided information along with a cover letter summarizing the job, diffraction patterns and EDS spectra for selected particles, an evaluation sheet for each chemistry, and some limited photographs of the particles observed in the microscope.

All data were produced as scanned images of count sheets, diffraction pattern computer screen dumps, chemistries, and the evaluations of the chemistries. All of the pages were stored in a pdf format.

1.2.1 Count Sheets

Process: The count sheet data on the CDs were converted to spreadsheets using a program called Able2Extract. After conversion, the data were manually verified for each count sheet by comparing the converted data with the pdf sheet. All of the columns on the count sheet (except those listed as "Count Categories") were reviewed and verified; the "Count Categories" column received a cursory review. Any revision to the converted data was made on-line to the appropriate spreadsheet. Only the Lab/Cor project files listed as "direct preparation" were converted - the indirect preparation files were not evaluated because the indirect procedure is known to modify the size and number of mineral particles. A final data entry validation step (performed to ensure the data were properly entered) included the examination of the data for all particles that fit into one of five categories and the correction of observed errors: 1) missing values; 2) data entries inconsistent with ISO 10312 counting rules; 3) particles with aspect ratios less than 3:1; 4) amphibole particles thinner than 0.1 μm or wider than 3 μm; and 5) particles with lengths shorter than 0.5 μm or longer than 20 μm. This action ensured that the data evaluated was consistent with the analytical method and that data values far from the average were correctly transcribed.

Evaluations: RJLG evaluated the replicate and duplicate counts performed by Lab/Cor and compared them to accepted performance criteria for such counts.

RJLG graphed the particle size data in various formats and compared those expected for asbestos and nonasbestos particle populations.

1.2.2 Diffraction Patterns

Process: The diffraction (SAED) pattern data were manually entered into spreadsheets listing the fiber identification, fiber dimensions, reported zone, and highlighted matching zone for each diffraction pattern. All data entries were verified by a second person with any corrections made on-line to the spreadsheet.

Evaluations: RJLG evaluated the SAED patterns provided by Lab/Cor, re-measured the patterns when possible, and compared the results of RJLG's SAED pattern identification with Lab/Cor's results.

RJLG evaluated the crystal directions represented by the orientations of the patterns to determine if they were consistent with an asbestiform population.

1.2.3 Spectra

Process: The reported chemistry data (EDS) were manually entered into a spreadsheet. The information for each EDS included a particle identification, the reported chemistry (oxide percentages), assigned atoms, and mineral identification. All data entries were verified by a second person with any corrections made on-line to the spreadsheet.

Evaluations: RJLG compared the calculated aluminum concentrations with values found in the literature for asbestiform and nonasbestiform calcic amphiboles.

RJLG reviewed the quantitative output of Lab/Cor's conversion of the observed chemistry (weight percent of oxides) to the number of atoms in an amphibole mineral used to determine the IMA classification⁴¹ for the particle.

RJLG evaluated the reliability of Lab/Cor's EDS results for individual particles by considering the possibility that the silicon content was overestimated.

RJLG plotted the reported concentrations for aluminum on a ternary plot and compared those concentrations with the upper limit on the concentration of aluminum in asbestiform amphiboles reported in Verkouteren and Wylie⁴².

1.2.4 Additional Reviews Performed

The documents generated during RJLG's review of the EPA data are found in Attachment C-1.2.4a. The data produced by Lab/Cor and evaluated by RJLG is Attachment C-1.24b. RJLG's results of re-measurement of Lab/Cor's SAED patterns are Attachment C-1.2.4c. The data resulting from evaluating the effect of the normalization of EDS data when performing a standardless analysis as was done by Lab/Cor is Attachment C-1.2.4d. As seen in the following, RJLG found the Lab/Cor data to have significant uncertainties, measurement errors and systematic bias.

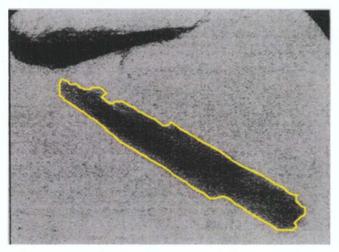
Morphology

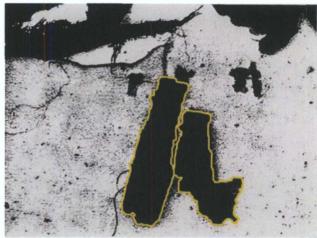
Lab/Cor provided forty-four (44) photographs out of nearly two thousand four hundred (2,400) particles analyzed. While this is an extremely limited number of photographs given the number of particles in the data set, it would be expected that they are at least

⁴¹ Leake, B.E. et al. (1997). Nomenclature of the amphiboles: Report of the subcommittee on amphiboles of the International Mineralogical Association, Commission on New Minerals and Mineral Names: Canadian Mineralogist, 35, p. 219-246.

⁴² J.R. Verkouteren, A. G. Wylie (2002). "Anomalous optical properties of fibrous tremolite, actinolite, and ferro-actinolite," American Mineralogist, 87, p. 1090-1095.

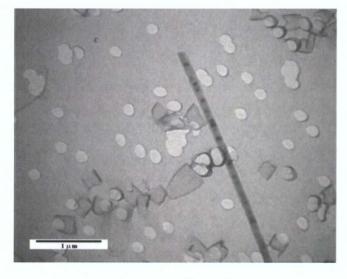
representative of the particle population. The Lab/Cor photographs are found in Attachment C-1.2.4e. Each particle has been highlighted to illustrate the shape of the particle. As seen in the attachment, and in the exemplar in Figure 10 below, many of the particle images Lab/Cor collected do not have parallel sides as required in the ISO 10312 method.⁴³

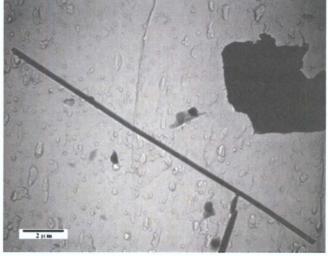




Lab/Cor Image of Counted Particle from El Dorado Hills

Lab/Cor Image of Counted Particle from El Dorado Hills





Jamestown Tremolite

Korean Tremolite

Figure 10. A comparison of the particles observed by Lab/Cor with asbestiform fibers observed in Jamestown (left) and Korean (right) tremolite samples.

⁴³ ISO states: "fibre: An elongated particle which has parallel or stepped sides." The "stepped sides" was added to the definition at the request of Dr. E. Chatfield to accommodate his observations of grunerite which can have parting planes within a structure. However, the grunerite fibrils must still have parallel sides.

TEM methods give some latitude in the definition of parallel sides, (e.g., in most laboratories, a long thin, particle having curvature or showing flexibility need not have as stringent a definition of parallel sides as a short straight particle) but the Lab/Cor particles do not meet the standards used by experienced laboratories or those intended by the authors of the methods as illustrated by the comparison with the photo of amphibole asbestos (see Figure 6) from the EPA Asbestos Fiber Atlas.⁴⁴ It is this variation that likely gave rise to the significant reproducibility issues in the verified counting data produced by Lab/Cor, since using only a minimum 3:1 aspect ratio gives wide latitude in the shape of the particle included in the count. RJLG recorded SEM and TEM photos of particles from the El Dorado Hills soil and airborne particles generated by use of the EPA Superfund elutriator method from those same samples.

SAED Analyses

RJLG examined SAED data from 408 particles analyzed by Lab/Cor (both direct and indirect preparation). Table 1 illustrates the comparison of RJLG's evaluations with Lab/Cor's evaluations.

	# Samples	%
Agreement	7	1.7%
Similar Results But Equal or Better Mineralogical Interpretation	90	22.1%
No SAED	1	0.2%
Fits Outside Acceptable Error	8	2.0%
Poor Measurements and/or Questionable Camera Constant	6	1.5%
Different Results	66	16.2%
Unable To Measure	230	56.4%
Total	408	100.0%

Complete agreement with the Lab/Cor mineral identification was obtained in only 7 (1.7%) of the 408 particles. For an additional 90 particles, RJLG's evaluation of Lab/Cor's provided patterns resulted in similar but equal or better mineralogical interpretations. Therefore, Lab/Cor properly identified no more than and probably significantly fewer than 25% of the particles.

A total of 230 (56.4%) of the patterns could not be interpreted based upon the supplied data. RJLG's analysis shows that 66 (16.2%) of the SAED patterns should have been interpreted as a result different than Lab/Cor's call. An additional 8 (2.0%) of Lab/Cor's

⁴⁴ P. K. Mueller, A. E. Alcocer, R. L. Stanley, and G. R. Smith (1975). "Asbestos Fiber Atlas." U. S. Environmental Protection Agency, EPA-650/2-75-036.

reported results exhibited numerical fits that were outside the acceptable error range for Lab/Cor's call. Another 6 particles exhibited poor measurements and/or questionable camera constants, and no SAED pattern was provided for one particle.

In summary, RJLG's evaluation of the provided SAED patterns found unambiguous agreement with Lab/Cor's reported call in fewer than 2% of the collected diffraction patterns.

Nonasbestos minerals were reported by Lab/Cor as asbestos. Attachment C-1.2.4f contains the Lab/Cor data for those samples where hornblende particles were counted as asbestos.

Lab/Cor underestimated the number of hornblende identifications in the SAED pattern analysis. Figure 11 shows an output from the Lab/Cor SAED pattern analysis software that compares the fit of actinolite and hornblende with lower errors reported for hornblende. Lab/Cor identified this particle and some twenty-five additional particles as actinolite (see Attachment C-1.2.4g), even though the chemical analysis contained significant aluminum and the SAED pattern suggested hornblende was the best match.

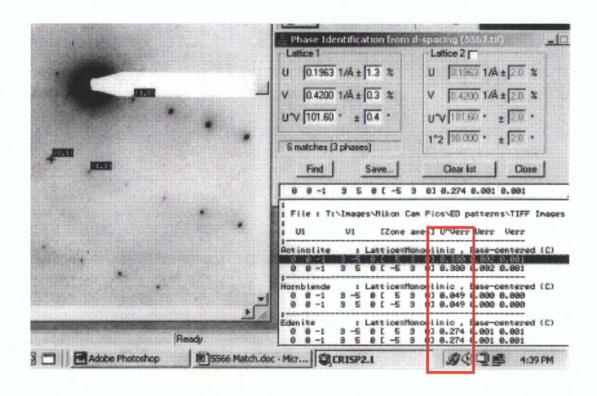


Figure 11. An example of the diffraction pattern measurement performed by Lab/Cor.

The red box shows the error associated with fitting the measured values to reference values. The smaller the error, the better the fit to the reference standard. In this instance, Hornblende is shown to be a better fit than Actinolite.

Approximately 3% of the Lab/Cor particles identified as amphibole had SAED measurements that were inconsistent with actinolite or tremolite. For example, Lab/Cor reported thirteen particles as actinolite or tremolite that had SAED patterns with a lattice spacing between ~ 9.9 and 12.2 angstroms. The high end of this range is as much as ~ 3 angstroms from the typical ~ 9.0 to 9.3 angstrom values characteristic of amphiboles. The patterns that were found to have lattice spacing values inconsistent with actinolite or tremolite are found in Attachment C-1.2.4h.

Lab/Cor inappropriately limited the search criteria in the SAED analysis. Approximately 30% of the particles identified as actinolite by Lab/Cor are equally or more likely to be hornblende or pyroxene. For example, several particles had pattern measurements reflecting a \sim 5.5 to 6.9 angstrom lattice spacing but were identified as actinolite. These measurements are inconsistent with actinolite but are consistent with pyroxenes (see Attachment C-1.2.4i).

Measurement errors were identified in 15% of Lab/Cor particles. As shown is Attachment C-1.2.4j, re-measurement of Lab/Cor SAED patterns, assuming the largest Lab/Cor d-spacing was accurate, resulted in a significant discrepancy. The origin of the Lab/Cor error was in the choice of spots to measure on the SAED pattern, which often did not measure across a sufficient number of unit cells, thereby maximizing measurement error.

EDXA Analyses

Lab/Cor and RJLG did not differ on the estimated atom concentrations used in making the Leake IMA classifications. The graph in Figure 12 shows the compositions of four major elements estimated by Lab/Cor versus those estimated by RJLG. No significant differences were observed between the Lab/Cor and RJLG analysis with respect to the concentrations of metals cations. The differences are in the mineral assignments.

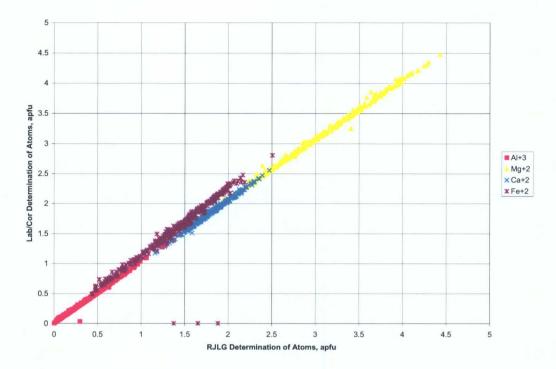


Figure 12. A comparison of the number of atoms for four elements calculated by RJLG and Lab/Cor. The data are based on the quantitative EDS reported by Lab/Cor. The data are in agreement in the number of atoms with minor variations in iron content due to the assumed valence state of iron.

The elemental analysis performed by Lab/Cor to determine the IMA mineral classification used normalized energy dispersive spectroscopy (EDS) data. Normalization of elemental concentrations performed as part of an EDS analysis is known to result in an overestimation of the silicon concentration in silicate minerals. Such a difference in the silicon concentration has a dramatic effect when classifying amphibole mineral species using IMA methodology. RJLG evaluated the Lab/Cor results to estimate the potential effects of errors in silicon concentrations and found that Lab/Cor may have underestimated the number of particles identified as hornblende in El Dorado by as much as 25%. Thus there is a high degree of uncertainty in the mineral assignments made by Lab/Cor.

1.2.5 Documentation of Additional Reviews

Reference samples of Naturally Occurring Asbestos from El Dorado Hills, San Andreas and Jamestown were collected and compared with the amphiboles in the soil from El Dorado Hills. Naturally occurring asbestos (NOA) specimens (tremolite) collected from an outcrop near one of the El Dorado Hills test sites and from two other locations in California were analyzed by RJLG.

The NOA specimens analyzed by RJLG had the characteristics of asbestos as described in the EPA QAPP for El Dorado Hills, and the characteristics recognized in all regulations

and methods of analysis, as diagnostic of asbestos. Specifically these specimens included bundles of fine, flexible, readily separable fibers. Particles photographed by Lab/Cor showed none of these characteristics. (Attachment C-1.2.4e) Each of the NOA samples analyzed by RJLG had both a population of asbestos and nonasbestos particles as defined by Campbell, Wylie, 45 and others.

NOA from El Dorado is tremolite asbestos, not actinolite or hornblende. The NOA specimens collected by RJLG in California, including El Dorado Hills, had the composition of tremolite and the fibers had no significant aluminum or iron concentration. The particles in the specimens collected in California have different shapes and chemistry than those particles reported as amphiboles by Lab/Cor, of which 80 percent contained a significant amount of aluminum or iron.

Natural amphibole asbestos occurrences have been the subject of debate in El Dorado County for nearly a decade. Veins of asbestos that outcropped locally have been identified, excavated, and covered. It is good laboratory and field practice to obtain and analyze reference materials particularly in complex mineral environments. Had EPA obtained, and Lab/Cor analyzed, material specimens from the known asbestos veins that outcropped locally in El Dorado County, EPA would have observed that the particles Lab/Cor reported in the samples from the EPA test sites were not consistent with asbestiform specimens from the region.

2.0 Soil Samples - Preparation Methodology

[EPA Request] Please list the soil preparation methods the R.J. Lee Group [sic] used to prepare splits of the EPA soil samples for analysis. A complete response to this inquiry will include information on whether a microscopic/stereoscopic analysis of the soil samples was conducted prior to any sample handling or preparation, drying times, moisture content, grinding (types and brands of grinders), sieving (sizes), and any other information required to provide a complete description of the preparation procedure used by the R.J. Lee Group [sic] for splits of the EPA soil samples.

RJLG received 23 soil samples from Youngdahl Consulting Group on August 26, 2005 (Fed Ex shipment 791183164750). These samples were identified as split samples that had been provided to Youngdahl by Ecology & Environment, Inc. RJLG did not split these samples or perform an examination of the samples prior to any sample handling or preparation. Upon receipt, the samples were assigned RJLG sample numbers and forwarded to our optical laboratory for analyses (see section 3.0).

⁴⁵ A.G. Wylie (1990). "Discriminating Amphibole Cleavage Fragments from Asbestos: Rationale and Methodology ", Exposure Assessment and Control Asbestos/Other Fibrous Material, p. 1065 – 1069.

RJLG prepared the soil samples for analysis and performed PLM and X-ray diffraction (XRD) analysis for asbestos in accordance with the published method.⁴⁶ RJLG performed elutriation tests on selected soil samples in accordance with the Berman-Kolk method.⁴⁷ RJLG prepared and analyzed TEM samples from four of the soil samples in accordance with published methods.

RJLG identified and photographed particles having a length to width ratio equal to or greater than 3:1 in the SEM 48 and TEM.

RJLG prepared samples for and performed Computer-controlled Scanning Electron Microscopy (CCSEM) on soil samples.⁴⁹

2.1 Drying Times

The soil samples received were visibly dry and therefore were not further dried prior to analyses. All results are reported on an "as-received" basis.

2.2 Grinding

The samples were not ground prior to analysis by RJLG in order to homogenize or otherwise produce a uniform particle size.

2.3 Sieving

On a follow-up analysis of soil sample NYT-SJ3-100804-FG2, the sample was dry screened using a 120 mesh (125 μ m) sieve (from Gilson Company, Inc.). The screening was performed in general accordance with ASTM D4749.⁵⁰

2.4 Other

Selected samples were also analyzed by SEM 48 or by XRD in accordance with published methods (see section 3.0). Prior to submitting the samples for these analyses, the samples were subdivided in general accordance with ASTM C702. 51

⁴⁶ R. L. Perkins, and B. W. Harvey (1993). "Method for the Determination of Asbestos in Bulk Building Materials", U.S. Environmental Protection Agency, EPA/600/R-93/116, July 1993.

⁴⁷ W. Berman and A. Kolk (2000). Modified Elutriator Method for the Determination of Asbestos in Soils and Bulk Materials, Revision 1, May 23, 2000.

⁴⁸ ISO 14966, Ambient Air – Determination of numerical concentration of inorganic fibrous particles – scanning electron microscopy method.

⁴⁹ ASTM (2001). "Standard Guide for Gunshot Residue Analysis by Scanning Electron Microscopy/Energy-Dispersive Spectroscopy", ASTM E1588. The method describes the general procedures to follow for CCSEM analyses.

⁵⁰ ASTM (2002). Standard Test Method for Performing the Sieve Analysis of Coal and Designating Coal Size, D4749.

⁵¹ ASTM C702 Standard Practice for Reducing Samples of Aggregate to Testing Size.

3.0 Soil Samples - Analytical Methodology

[EPA Request] Please list the analytical techniques (including full method name and reference number) that the R.J. Lee Group used to evaluate splits of the EPA soil samples. Please provide all documents generated as a result of that analysis including: laboratory count sheets, laboratory notes and logbook pages, sketches, images, chain-of-custody forms and other sample tracking sheets. Also include the same information for any and all QC samples analyzed along with the investigative samples, all required calibrations, and any other technical notes generated during the analyses of splits of the EPA soil samples. Please include a general description of the instruments (including make and model) used in the analyses and provide the analysts' names and qualifications to perform an analyses for each of the analytical/preparation techniques employed. Regardless of method, please provide laboratory count sheets and a full description of all exceptions or modifications to the analytic techniques.

The soil samples were evaluated using the procedures described in the EPA method.⁵² The SEM analyses of the samples were performed in general accordance with the analytical portion of ISO 14966.⁵³ The weight percent of the fine soil particulate was determined in general accordance with the analytical portion of ASTM D5756.⁵⁴ The soil samples were also evaluated using the Berman-Kolk elutriator method for releasable particulate.⁴⁷

The analytical methods used are included in Attachment C-3.0. The supporting documentation is described in the following sections.

3.1 Sample Analyses

Laboratory data are described in section 3.2. Hornblende was the only amphibole mineral identified in the soil samples. RJLG compared the chemistry and appearance of the particles found in the soil samples with asbestos fibers from two samples of amphibole asbestos collected in California, including one from El Dorado County.

3.2 Laboratory Count Sheets

The laboratory data for PLM analyses (count sheets and images) are included in Attachment C-3.2a. The laboratory data for SEM analyses (count sheets and images) are included in Attachment C-3.2b. The laboratory data for TEM analyses (count sheets, images, EDS and SAED) are included in Attachment C-3.2c. The data from the CCSEM analysis are in Attachment C-3.2d. The laboratory data for the XRD analyses are in the

⁵² Interim Method for the Determination of Asbestos in Bulk Insulation Samples, 40 CFR Part 763, Appendix E to Subpart E,

⁵³ International Organization for Standardization (2002). "Ambient air. Determination of numerical concentration of inorganic fibrous particles. Scanning electron microscopy method", International Organization for Standardization, Geneva, Switzerland, ISO 14966:2002(E), 11-15-2002. Updated Edition.

⁵⁴ American Society For Testing And Materials. "Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Mass Concentration," D 5756-95, Annual Book of ASTM Standards, 1995, pp. 1-13.

Attachment C-3.2e. Laboratory data resulting from additional evaluations described in this document and in Exhibit A⁵⁵ and Exhibit B⁵⁶ are also included in these attachments.

3.3 Laboratory Notes and Logbooks

Other than the notes recorded on the count sheets included in Section 3.2, there are no laboratory notes related to the subject samples. Logbooks are not used in the PLM and TEM laboratories as samples are tracked electronically. Appropriate logbook pages from the XRD and SEM laboratories are in Attachment C-3.3.

3.4 Images

Photographs of various particles are included in the attachments described in Section 3.2. The photographs include those recorded using PLM, SEM and TEM.

3.5 Chains-of-Custody

The chain-of-custody form transferring the split soil samples to RJLG is in Attachment C-3.5a. The split samples remain in RJLG's possession.

3.6 Sample Tracking

Sample tracking is accomplished electronically.

3.7 Personal Qualifications

Personnel qualifications are included in Attachment C-3.7.

QA/QC Procedures

[EPA Request] Please submit all documents regarding the R.J. Lee Group's [sic] quality assurance/quality control ("QA/QC") procedures for asbestos analyses. Please include in this response information regarding the processes for and results of laboratory monitoring, sample preparation, laboratory analysis, data management, laboratory certifications, internal and external report review processes, and internal and external peer review processes. Please also include all Standard Operating Procedures ("SOPs"), Laboratory Quality Assurance Plans or other information relevant to or generated during the R.J. Lee Group's [sic] analyses of the EPA soil and air samples.

RJLG operates under an extensive Quality Assurance/Quality Control plan that has been thoroughly reviewed by NVLAP, AIHA, PA DEP, NY ELAP, CA ELAP and the US EPA.

⁵⁵ Response of RJ Lee Group to the United States Environmental Protection Agency Region IX Response (dated April 20, 2006) to the November 2005 National Stone, Sand & Gravel Association Report Prepared by the R.J. Lee Group, Inc [sic] "Evaluation of EPA's Analytical Data from the El Dorado Hills Asbestos Evaluation Project", July 2006.

⁵⁶ Response of RJ Lee Group to Mr. Meeker's Letter (undated) to Dr. Vicki Barber, El Dorado Hills School District, regarding Evaluation of EPA's Analytical Data from the El Dorado Hills Asbestos Evaluation Project, July, 2006.

RJLG's QA/QC plan is a company proprietary document that EPA reviewed during its inspection of our laboratory in 2004. The EPA is invited to visit the laboratory at a mutually agreeable time to review this document again if needed.

4.1 Certifications

RJLG maintains laboratory certifications/accreditations in a number of venues. Copies of relevant certifications are in Attachment C-4.1.

4.2 Laboratory Standard Operating Procedures

RJLG's Laboratory SOPs are company proprietary documents that EPA reviewed during its inspection of our laboratory in 2004. The EPA is invited to visit the laboratory at a mutually agreeable time to review these documents again if needed.

4.3 Laboratory Monitoring

As part of its QA/QC program, RJLG performs routine monitoring of its laboratory spaces for possible asbestos contamination. Copies of the monitoring most relevant to the EDH project are in Attachment C-4.3.

5.0 Review Documents

[EPA Request] Please identify the supporting documents or information that were made available to the Association or the three outside reviewers of the R.J. Lee Report for their respective review of the quality of the R.J. Lee Report or the quality of the data supporting the R.J. Lee Report. In addition to this statement, please also provide the documents or information in the statement that are not otherwise provided in response to this Information Request. Documents responsive to this request may include: SOPs; QA/QC procedures; performance evaluation samples; third party audits; notes; analytical techniques; literature cited in the R.J. Lee Report; other scientific literature; R.J. Lee Group [sic] procedures and documentation; written communications, phone logs; and electronic mail.

A draft version of the report was circulated among three outside reviewers (Wylie, Langer, Ross) and among the NSSGA. No supporting documentation or other publications were requested by the various reviewers at that time. Any questions raised by the reviewers were answered by telephone. A copy of the draft report that was sent to the reviewers is in Attachment C-5.0 along with the reviewer responses. Procedures governing project review are described in RJLG QA/QC procedures.

5.1 Third Party Audits

RJLG is routinely audited by outside personnel. The audits are usually associated with renewal of a laboratory certification. Among the groups that have audited RJLG are NVLAP and AIHA. Appropriate to this response is an audit performed of RJLG's laboratory by EPA in 2004, noting:

". . . auditors were hard pressed to find any deficiencies at this corporate asbestos testing laboratory. This facility could easily be considered among the most capable of those laboratories audited and approved by ASB for Superfund asbestos testing needs. All staff, from sample log-in personnel to microscopists, are extremely knowledgeable and experienced. Raw data is managed by instrument dedicated data entry PDAs coupled to a LIMS server which makes reporting of incomplete results almost impossible. The facility also has a very good Quality Assurance Program designed to encompass all operations at the corporate, as well as, regional facility locations."

5.2 Cited Literature

Copies of the literature cited in RJLG's November 2005 report are in Attachment C-5.2. Copies of the relevant literature were not provided to the reviewers at that time as they are very knowledgeable about the subject in question and already had access to the relevant literature.

5.3 Communications

A search of e-mails and records of telephone conversations was made; no such records relevant to this question were found. No letters were written as part of this project. There were phone calls between the reviewers and RJLG, but no records of these conversations were made. RJLG does not require the retention of telephone logs.

6.0 RILG OA/OC Procedures

[EPA Request] Please provide a statement describing the R.J. Lee Group's [sic] QA/QC procedures for each analytic technique to ensure consistency in measurements of asbestos with particulate loading or asbestos in soil (e.g., structure identification, mineral identification, diffraction patterns, reported concentrations, etc.) within the laboratory and among two or more analysts examining any respective sample. The statement should include, for each analytical technique relevant to the R.J. Lee Report, the R.J. Lee Group's variability rate among its analysts for samples of asbestos with particulate loading or asbestos in soil. Please also provide all documents that support the determination or assessment of the variability rates.

The requested procedures were reviewed by the EPA in their 2004 audit of RJLG's Monroeville laboratory. The variability of our analysts is within the accepted variability rate of analysts as determined by the accrediting agencies. The various external audits document review of RJLG's analytical and QA/QC procedures as noted in the recent EPA audit of our Monroeville laboratory.

RJLG is an accredited laboratory that performs analyses for a wide variety of clients, including environmental engineering firms, industrial hygienists, federal, state, and local governmental agencies, and individual building owners. As such, RJLG's QA/QC procedures have been rigorously evaluated by outside agencies, including the US EPA and

our analytical capabilities meet and exceed the requirements of the various accrediting agencies.

The testing performed by RJLG on the soil samples from EDH was a multi-phase test program that encompassed a variety of analytical procedures. The analytical results from each technique support the results of the other techniques performed on the samples – no amphibole asbestos was observed in the split soil samples examined by RJLG, but varying amounts of hornblende were observed in these samples.

7.0 Issues Related to Asbestos Identification

[EPA Request] Please provide all documents that support or explain the following issues raised by the R.J. Lee [sic] Report:

7.1 Characteristics of Asbestos Fibers

[EPA Request] The character of fibers counted as asbestos fibers in the R.J. Lee Report, specifically those relevant to establishing limitations on width or the exclusion of "cleavage fragments," considering the May 30, 2003 REPORT ON THE PEER CONSULTATION WORKSHOP TO DISCUSS A PROPOSED PROTOCOL TO ASSESS ASBESTOS-RELATED RISK, which addresses protocols on assessing asbestos related risks under the Berman-Crump method, and which recommended counting "cleavage fragments" that have equal durability and dimension as asbestos fibers, and recommended, to account for inhalation through the mouth, counting fibers up to 1.5 microns in width.

RJLG did not consider the individual fibers reported by Lab/Cor as asbestos or nonasbestos in the review of the El Dorado Hills Report. Rather, RJLG examined the group of reported fibers to evaluate issues related to aluminum content and particle size. RJLG found that the aluminum content of the amphibole particles analyzed by Lab/Cor was higher than that reported in the scientific literature for calcic-amphibole asbestos fibers and also determined that the length/width distribution of the reported fibers was not that of an asbestos population.

RJLG addresses the key questions relevant to EPA Region 9's question below:

What fibers should be counted and considered in the risk assessment? If EPA Region 9 and ATSDR intend to base the risk assessment on its QAPP and on IRIS, the answer to the question is: the asbestos concentration of airborne samples is determined by measuring the number of asbestiform fibers meeting the counting rules of the method employed to evaluate the risk under IRIS. In the case of environmental samples, this requires the use of phase contrast microscopy PCM (NIOSH 7400) as the base analysis under which the fiber concentration is determined and TEM (NIOSH 7402) to determine the proportion of fibers counted that are asbestos. In this instance the upper limit on the width of asbestos fibers to be counted is 3 μ m.

EPA has recognized the limitations of IRIS and has begun a full review of IRIS with the intent of updating/revising the procedure. FPA Region 9 and ATSDR intend to use the more modern risk analysis written by Berman and Crump8, the answer to the question is: only fibers whose width (whether or not they are asbestiform) is less than $0.4~\mu m$ and whose length is longer than $10~\mu m$ would be counted.

If there is a concern about the potential interference in the asbestos count and/or risk presented by cleavage fragments, the analyses could have been done as the New Jersey Department of Health and EPA Region 2 did in the Southdown Study. In that case, the answer is: Both asbestiform fibers and cleavage fragments should be identified, photographed and counted, enabling a formal risk assessment for the two populations of particles using the criteria deemed appropriate by the risk experts.

The size distribution of the measured particle population is inconsistent with that of asbestos particles; therefore, the use of the IRIS risk model (which is based on exposure to commercial asbestos) is inappropriate. As a result, risk estimates for the El Dorado data set should be based on the model outlined in the Berman-Crump Technical Support Document for a Protocol to Assess Asbestos-Related Risk⁸, which is far more applicable to mixed dust environments than is IRIS because epidemiology studies from mixed dust environments were included in the method development.

The question about the character of fibers reported as asbestos, however, goes beyond the El Dorado data set (whose interpretation is limited by the nature of the data collected) to a more general question of what is asbestos.

What dimensions should be counted? Asbestos is a recognized carcinogen for which specific models to estimate the health risk resulting from exposures have been developed. As pointed out in the Berman-Crump technical support document⁵⁸ published as a follow-up to the peer consultation report,⁵⁹ to be valid, risks estimated using such models must meet two criteria: 1) asbestos must be measured in a comparable manner in the two environments; and 2) such measurements must remain reasonably proportional to the characteristics of exposure that contribute to risk. This means that fibers that have the dimensions of the asbestos fibers to which the original study cohort was exposed should be counted in the El Dorado Hills Study.

The technical support document states that the optimal exposure index assigns a single potency to fibers that are longer than 10 μ m and less than 0.4 μ m in width and zero potency to fibers outside these dimensions. The potency index would be different for lung

⁵⁷ EPA 2006. Federal Register, volume 71, p. 9333 – 9336, February 23, 2006.

⁵⁸ D. W. Berman and K. Crump (2003). Technical support document for a protocol to assess asbestos-related risk," U.S. Environmental Protection Agency, Revision of original from September 4, 2001, Peer-reviewed consultation held in San Francisco on February 25-26, 2003.

⁵⁹ USEPA (U.S. Environmental Protection Agency) (2003). Report on the Peer Consultation Workshop to Discuss a Proposed Protocol to Assess Asbestos-Related Risk, Final Report. Office of Solid Waste and Emergency Response, Washington D.C.

cancer than for mesothelioma. <u>This means that the concentration of long thin fibers is the most relevant to describing the potential hazard of a dust.</u>

The peer consultation panel generally accepted these concepts with some qualifications and proposals for additional study. One of the suggestions for consideration was that thicker fibers (0.5 μm to 1.5 μm in width) not be excluded when considering lung cancer, although it was recognized that they have a much lower probability of penetrating into the lung. Another suggestion was that only thin (<0.5 μm) fibers be included for mesothelioma, but that fibers from 5 to 10 μm long be included in the index for lung cancer.

Another panel suggestion was that lacking other specific information, nonasbestos amphibole (cleavage) fragments of the same size and dimension as asbestos fibers be treated as though they were as potent as asbestos. One panelist emphasized that there are distinct morphological and chemical differences between naturally formed asbestos fibers and fragments whose surfaces were produced by fracture. Members of the panel noted that numerous studies have shown no asbestos disease amongst miners heavily exposed to nonasbestos amphiboles including the New York Talc miners, 60 gold miners in Homestake, South Dakota and Minnesota taconite 2 ore workers. This means that while the panel voiced opinions on the significance of cleavage fragments, no peer-reviewed EPA, OSHA, NIOSH, or ATSDR document has found that cleavage fragments cause asbestos disease.

The panel emphasized the need to reconcile the disparity between the Quebec miners and South Carolina chrysotile textile worker exposures. The recent NIOSH reanalysis of the South Carolina⁹ data reinforces the premise that the presence of long thin (>40 µm long, <0.3 µm wide) asbestos fibers in an aerosol is the most important measure of the potency of the dust.

The clear and unambiguous message in the health and risk data is that long, thin airborne fibers are a prerequisite for asbestos disease – absent long, thin airborne fibers, the data does not indicate any significant potency. The second important consideration is to ensure that particles with those size characteristics are reliably measured. While there is no specific evidence that long, thin cleavage fragments (>10 μ m long, thinner than 0.5 μ m) present a risk of asbestos disease, they are a rare occurrence and prudent public policy may indicate they should be counted as asbestos.

Should cleavage fragments be counted as asbestos? EPA Region 9 set forth a project plan specifically in response to concerns voiced by a citizen over potential exposures to

⁶⁰ Y. Honda, C. Beall, E. Delzell, K. Oestenstad, I. Brill, and R. Matthews (2002). "Mortality Among Workers at a Talc Mining and Milling Facility", Ann. Occup. Hyg., <u>46</u>, p. 575-585.

⁶¹ K. Steenland and D. Brown (1995). "Mortality Study of Gold Miners Exposed to Silica and Nonasbestiform Amphibole Minerals: An Update with 14 more years of Follow-Up", American Journal of Industrial Medicine, <u>27</u>, p. 217-229.

⁶² W. C. Cooper, O. Wong, L.S. Trent, and F. Harris (1992). "An Updated Study of Taconite Miners and Millers Exposed to Silica and Non-Asbestiform Amphiboles." JOM, <u>34</u>, p. 1173-1180.

asbestos in and around the El Dorado Hills School District. EPA Region 9, in conformance with past EPA practice as well as that of other agencies, recognized the mineralogical and geological vocabulary and defined asbestos for the purpose of the study as "fiber bundles made up of extremely long and thin fibers that are readily separated from one another." ⁶³ EPA Region 9 did not differentiate between regulated asbestos and non-regulated asbestiform minerals.

EPA Region 9's original project goals and definitions were consistent with the known characteristics of asbestos, both mineralogically and as a recognized hazardous substance. Counting nonasbestos particles as if they were asbestos is a deviation from the project plan.

The character of fibers counted as asbestos by a laboratory should be that of asbestos unless a specific exception to the method is noted. In an environmental analysis all particles meeting the method counting criteria are identified, measured, and reported appropriately as amphibole, amphibole asbestos, serpentine, chrysotile, or other mineral. These are the requirements of the Yamate and the NIOSH 7402 methods. Neither places a restriction on the counting of structures with diameters between 0.5 and 1.5 μ m. The answer to EPA Region 9's question is that all particles meeting the counting criteria specified by the method should be counted, reported and classified as either asbestos or nonasbestos. If risk criteria implicating nonasbestos amphiboles in disease are identified, their concentrations should be subject to a formal risk assessment as has been done for asbestos in the IRIS method. However, given nonasbestos particles do not, in general, have equiaxed cross-sections and have different aerodynamic qualities and deposition characteristics than their asbestiform counterparts, the risk analysis of nonasbestos particles should be independent of any asbestos risk analysis.

It is not a laboratory decision to determine whether or not cleavage fragments are to be counted from a risk perspective. It is a laboratory responsibility to certify that the particles counted and reported as asbestos are in fact asbestos. Lab/Cor signed a contract to identify and count asbestos particles and signed reports indicating they had found asbestos when they did not.

At a higher policy level, EPA Region 9 used their authority under CERCLA to assess the significance of potential asbestos exposures. Given the recognized differences between asbestos and nonasbestos amphiboles, both at the mineralogical and regulatory levels, any response action ought to be based on measurements of asbestos as it was defined by the QAPP.

Setting public policy by using the peer consultation report to bootstrap an equivalent asbestos exposure to mineral fragment exposure is inappropriate. Further bootstrapping the process by using risk estimates derived for long, thin commercial asbestos fibers to evaluate the significance of short, wide nonasbestos fragments is inappropriate.

⁶³ Ecology & Environment (2004). page ix.

7.2 Iron Valence State

[EPA Request] How the R.J. Lee Group [sic] in the R.J. Lee Report distinguished between the presence of Fe^{3+} and Fe^{2+} found in amphibole minerals when performing an Energy Dispersive Spectroscopy ("EDS" or "EDXA" in the R.J. Lee Report) analysis.

The identification of valence state for iron should be determined using Mössbauer spectroscopy or by wet chemistry as described in the literature.⁶⁴ No such data was produced by Lab/Cor; RJLG has not performed such an evaluation for the amphibole particles in the soil samples examined.

The valence state (Fe³⁺ or Fe²⁺) was evaluated in accordance with the procedure described in Leake, Appendix A, beginning with step 6.⁶⁵ As implemented, the procedure does not require the actual ratio when determining the name to assign to a mineral. If the "all ferrous iron" name and "all ferric iron" name are the same, there is no need to determine the actual ratio of iron valence states.

RJLG evaluated the names assigned to 341 actinolite/tremolite particles by assuming the observed iron to be either all Fe^{3+} or Fe^{2+} . Forty-seven (47) particles resulted in a different name (generally to a hornblende).

7.3 Clay Contamination of Amphibole Particles

[EPA Request] How the R.J. Lee Group [sic] in the R.J. Lee Report distinguished between the signal of an amphibole structure from the aluminum signal from aluminum-rich clay particles adhered to the amphibole structures when performing an EDS analysis.

RJLG analyzed selected soil samples by XRD and determined the aluminum-bearing minerals in the samples to be hornblende, chlorite, plagioclase feldspar, and muscovite/vermiculite.

As part of the limited electron microscopy (EM) analyses performed by RJLG, the selected soil samples were prepared for analysis using an indirect preparation procedure. During this procedure, the soil is suspended in a liquid and is agitated using ultrasonication. This procedure separates loosely adhering particles. The samples were carefully examined in the EM to determine if any particles were adhering to the target particle and, if so, whether the adhering particles would interfere with the analysis of the target particle. We determined that this was not an issue in our analyses. This is documented in the photographs taken during these analyses.

When evaluating the Lab/Cor data, RJLG accepted the reported data at face value. It is the obligation of the microscopist to obtain an EDS spectrum from a portion of the particle that is most representative and to avoid interferences from adhering particles.

⁶⁴ F. C. Hawthorne (1983). "Mössbauer Spectroscopy", The Canadian Mineralogist, <u>21</u>, p. 264-276.

⁶⁵ Leake, B.E. et al. (1997). Nomenclature of the amphiboles: Report of the subcommittee on amphiboles of the International Mineralogical Association, Commission on New Minerals and Mineral Names: Canadian Mineralogist, 35, p. 219-246.

Clay particles are generically alumino-silicates; their elemental composition is aluminum, silicon and other elements. As shown in Deer, et al,⁶⁶ the silcon:aluminum ratio for the atoms in clay minerals ranges from 1:1 for the kaolinite group, from 1:1 to 3:1 for the illite group, and from 2:1 to 6:1 for the montmorillonite group of clay minerals. For every atom of aluminum that must be accounted for from the clay mineral, at least one (and up to six) silicon atoms must also be accounted for from the clay minerals.

Thus, for there to be an exclusion of aluminum due to the presence of clay, there must also be an exclusion of silicon. This exclusion of silicon was apparently not done by Lab/Cor, therefore, one must conclude there was no interference in the EDS spectra due to the presence of adhering clay minerals.

7.4 Variation of SAED with Particle Size

[EPA Request] How the R.J. Lee Group [sic] in the R.J. Lee Report distinguished or otherwise considered in its comparison based on zone axis indices the EPA soil samples containing mixed-sized particulates from the reference of asbestos sample standards.

Tremolite-actinolite asbestos fibers have limited widths (0.1 to 0.3 μ m) but can vary in length from microns to inches. The length of the asbestos fiber will have no effect on the observed zone axis⁶⁷ pattern. Nonasbestos amphibole minerals, however, will display varying zone axes depending on the fracture characteristics and orientation of the particle in the microscope. This variation in zone axis is one of the defining characteristics of the nonasbestos minerals.

It is well known that amphibole asbestos fibers have a tendency to orient in a preferred manner. This has been reported by Lee,⁶⁸ Nord,⁶⁹ Yamate,⁷⁰ Ring,⁷¹ and Stewart,⁷² and is recognized in all TEM analytical methods as nearly diagnostic of asbestos. This preferred

⁶⁶ W. A. Deer, R. A. Howie, and J. Zussman (1962). <u>Rock-Forming Minerals: Vol 3, Sheet Silicates</u>, John Wiley and Sons, Inc., p. 191-257.

⁶⁷ A "zone axis" is a way of describing the common direction of the intersections of the faces of a crystal. As used here, the "zone axis" describes a particular selected area electron diffraction pattern.

⁶⁸ R. Lee, J. Lally, and R. Fisher (1977). "Identification and Counting of Mineral Fragments", proceedings of the Workshop on Asbestos: Definitions and Measurement Methods held at NBS, Gaithersburg, MD, July 1977. NBS Special Publication 506, pp. 387.

⁶⁹ G. Nord and R. Lee (2003). "Characterization of Fibrous Particles by Analytical Transmission Electron Microscopy", Contained within "Program and Abstracts for International Symposium on the Health Hazard Evaluation of Fibrous Particles Associated with Taconite and the Adjacent Duluth Complex ", March 30-April 1, 2003.

⁷⁰ Yamate, G., S. C. Agarwal, R. D. Gibbons (1984). "Methodology for the Measurement of Airborne Asbestos by Electron Microscopy." EPA Contract No. 68-02-3266, July 1984.

⁷¹ S.J. Ring (1981). Identification of Amphibole Fibers, Including Asbestos, Using Common Electron Diffraction Patterns. In Russell P.A. and Hutchings A.E. (Eds), Electron Microscopy and X-ray Applications to Environmental and Occupational Health Analysis, Vol. 2:175-198, Ann Arbor Science Publ., Inc.

⁷² Stewart, I. (1988). "Asbestos - Analytical Techniques Definitions and Mineralogy of Asbestos Basic Crystallography and Electron Diffraction." Presented at JEOL TEM training courses.

orientation gives rise to the so-called characteristic 0.53 nm row spacing referred to in EPA methods for TEM analysis of asbestos.⁹⁶ In contrast, nonasbestos amphiboles tend to have far greater variability in orientation and do not predominantly show the 0.53 nm spacing. Rather, as shown in Figure 13, the nonasbestos amphiboles in this study cluster around a different direction (110).

Examples of zone axis patterns for tremolite-actinolite are shown in the following Figure 13. The top two patterns in the figure are indicative of nonasbestiform amphiboles and the bottom two patterns are indicative of asbestiform amphiboles.

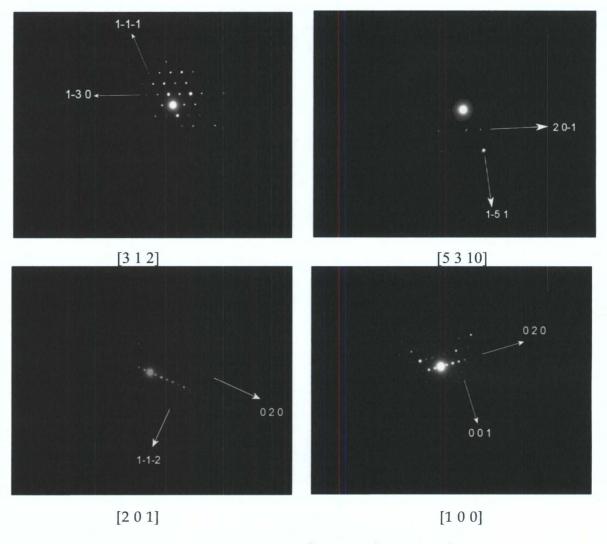


Figure 13. Zone axis patterns for tremolite-actinolite.

7.5 Different Morphologies of Amphibole Particles

[EPA Request] How, during a Polarized Light Microscopy ("PLM) analysis, the R.J. Lee Group [sic] in the R.J. Lee Report allowed for the presence of both asbestiform and nonasbestiform habits of the same mineral to be present in a rock or soil sample.

Within a geological setting where asbestos fibers are found, nonasbestos forms of the same mineral will always be observed. The converse is not true because specific geological conditions are needed for the development of asbestos fibers. RJLG "allows" for this occurrence by reporting the presence of all forms of the amphibole minerals. The characteristics of asbestos as described in the PLM methods, supplemented by the descriptions found in Campbell, 10 McCrone, 73 Wylie 74, and others, are used to identify a particle as asbestos.

7.6 Analysis of Amphibole Minerals

[EPA Request] Whether the asbestos amphibole fibers that the R.J. Lee Group [sic] counted for the R.J. Lee Report only included the six regulated asbestos mineral types that exhibit an asbestiform habit (>20:1 or 50:l aspect ratio) and exhibit parallel extinction.

When analyses are performed on commercial product material to determine conformance with various regulations, the asbestos types are limited to the six regulated minerals in accordance with the analytical methods and our laboratory certifications. For samples of raw materials, RJLG will not limit the reporting of asbestiform minerals to those that are regulated.

In the analyses of the El Dorado samples, RJLG applied the PLM methods as described in the analytical methods and Campbell,¹⁰ McCrone,⁷³ Wylie,⁸⁴ and others for additional descriptions of asbestos mineralogy. These analyses were not limited to the regulated asbestos types.

EPA contends that the mineral nomenclature complies with the Leake protocol.⁷⁵ RJLG has not, contrary to EPA statements (EPA page 5), challenged the assigned nomenclature but has noted that the amount of aluminum in the minerals precludes the formation of asbestos fibers.

Meeker² (page 3) suggests that a hornblende mineral (fluoro-edenite) that reportedly occurs as asbestos is proof that amphibole particles can contain significant quantities of aluminum and still be considered asbestos. Fluoro-edenite is not the same mineral as the actinolite/tremolite that is reported in the Lab/Cor data. It should be noted that "Edenitic compositions are rare in amphiboles, and their paucity might suggest a structural instability."⁷⁶

⁷³ W. C. McCrone (1980) The Asbestos Particle Atlas, Ann Arbor Science.

⁷⁴ A. G. Wylie, "Relationship Between the Growth Habit of Asbestos and the Dimensions of Asbestos Fibers", Mining Engineering, November 1988, p. 1036-1040.

⁷⁵ EPA, 2006, page 6.

⁷⁶ A. Gianfagna and R. Oberti (2001). Fluoro-edenite from Biancavilla (Catania, Sicily, Italy): Crystal chemistry of a new amphibole end-member, American Mineralogist, 86, p. 1489-1493.

The real issue is the aluminum content of the tremolite-actinolite amphibole minerals – the minerals at issue in El Dorado Hills. Deer et al⁷⁷ note (page 141) that in "most tremolite-actinolites, the replacement of Si by Al is small (<0.3 Al pfu)" and (page 182) that "Electron probe analyses showed that specimens that contain more than a very small amount of aluminum do not have asbestiform habit." Deer cites Dorling and Zussman⁷⁸ for the low aluminum content. Dorling and Zussman show (Figure 16 of their paper) that aluminum atoms in the asbestos samples analyzed were present at less than 0.1 apfu. The Dorling findings were supported by Verkouteren and Wylie⁷⁹ who showed 85% of their asbestos samples contained 0.1 Al apfu or less.

7.7 Parallel Extinction of Asbestos Fibers

[EPA Request] During a normal PLM analysis, whether the R.J. Lee Group [sic] in the R.J. Lee Report would consider parallel extinction to be a definitive indicator that an amphibole fiber is an asbestos fiber.

The use of parallel extinction is a defining characteristic of asbestos fibers as noted by Verkouteren and Wylie⁸⁰ and in the analytical protocols⁹⁷ when the fibers also exhibit other characteristics of asbestos. However, the observation of parallel extinction without other asbestos characteristics does not define a particle as asbestos. Asbestos is characterized by bundles of easily separated fibers, very thin fibers (less than $0.5~\mu m$), fibers showing curvature, and fibers with very high aspect ratios. When a population of particles does not exhibit any of these characteristics and does not show parallel extinction, the population of particles is clearly not asbestos.

The PLM methods state that asbestos fibers have parallel extinction while nonasbestos particles have oblique extinction. As noted in the PLM method used by the EPA (NIOSH 9002), tremolite-actinolite will have oblique extinction (10° - 20°) for fragments. As noted in OSHA ID-191 (section 3.5): "... cleavage fragments of the monoclinic amphiboles show inclined extinction under crossed polars with no compensator. Asbestos fibers usually show extinction at zero degrees or ambiguous extinction if any at all." The draft ASTM method (P236)81 that was circulated by NIST to all NVLAP laboratories states: tremolite

⁷⁷ W. A. Deer, R. A. Howie, and J. Zussman (1997). <u>Rock-Forming Minerals: Vol 2b Second Edition, Double Chain Silicates</u>, The Geological Society.

 $^{^{78}}$ M. Dorling and J. Zussman (1987). "Characteristics of asbestiform and non-asbestiform calcic amphiboles", Lithos, $\underline{20}$, p. 469-489.

⁷⁹ Verkouteren, J.R., and Wylie, A.G. (2000). "The tremolite - actinolite - ferro - actinolite series: Systematic relationships among cell parameters, composition, optical properties, and habit, and evidence of discontinuities", *American Mineralogist*, <u>85</u> p. 1239 – 1254.

⁸⁰ J. R. Verkouteren, A. G. Wylie (2002). "Anomalous optical properties of fibrous tremolite, actinolite, and ferro-actinolite," American Mineralogist, 87, p 1090-1095.

⁸¹ Proposed Test Method for Asbestos-Containing Materials by Polarized Light Microscopy, D-22, Proposal P236.

asbestos and actinolite asbestos has extinction "parallel in most fibers". The EU method⁸² (1997) states that "polarized light microscopy (PLM) can be used to exclude some elongated cleavage fragments on the basis of their non-parallel extinction angle" (page 13). As noted in EPA's own 1993 PLM method⁸³ refractive indices are to be measured on tremolite-actinolite when the fiber exhibits extinction at a zero degree orientation (page 15). Wylie,⁸⁴ Dorling and Zussman⁸⁵, and Verkouteren and Wylie⁸⁰ report that asbestos fibers have parallel extinction or, if too thin, anomalous extinction properties.

7.8 NIST SRM 1867a

[EPA Request] Whether asbestos fibers supplied by the National Institute of Standards and Technology ("NIST") Standard Reference Materials ("SRM") 1867 and 1867a, as referenced in the R.J. Lee Report, ever exhibit inclined extinction angles.

The National Institute of Standards and Technology (NIST) certificates accompanying Standard Reference Materials SRM 1867 and 1867a⁸⁶ indicate the tremolite and actinolite standards in each are from the same batches of material and indicate that "a small amount of material may be massive" (tremolite) or "a considerable amount of material may be massive" (actinolite). The certificates note these minerals are "mine-grade asbestos materials". The asbestos in each sample is described as having asbestiform characteristics: "Asbestiform: crystallizes with the habit of asbestos. These asbestos minerals possess properties such as long fiber length and high tensile strength. Under the light microscope, [some portion of]⁸⁷ these samples exhibit the asbestiform habit as defined by several of the following characteristics: 1) mean aspect ratios ranging from 20:1 to 100:1 or higher for fibers longer than 5 μ m; 2) very thin fibrils, usually less than 0.5 μ m in width; 3) parallel fibers occurring in bundles; 4) fiber bundles displaying splayed ends; 5) fibers in the form of thin needles; 6) matted masses of individual fibers, and 7) fibers showing curvature" (page 2).

Figure 16 illustrates the morphologies of particles found in the NIST SRM 1867a tremolite and compares those to tremolite found near San Andres, California. Both images were taken using a PLM at the same magnification. The highly fibrous nature of the California tremolite is evident compared with the NIST SRM 1867a tremolite.

⁸² Burdett, G., "Final report for R42:70: Quantitative measurement of asbestos and other fibres in bulk materials 1R/L/MF/98/02", Environmental Measurement Group, Health and Safety Laboratory (An agency of the Health and Safety Executive, Broad Lane, Sheffield, S37HQ, August 1998.

⁸³ R. L. Perkins, and B. W. Harvey (1993). "Method for the Determination of Asbestos in Bulk Building Materials", U.S. Environmental Protection Agency, EPA/600/R-93/116, July 1993.

⁸⁴ A. Wylie (1979). "Optical properties of the fibrous amphiboles", Ann NY Acad Sci, 330, p. 611-619.

⁸⁵ M. Dorling and J. Zussman (1987). "Characteristics of asbestiform and non-asbestiform calcic amphiboles", *Lithos*, <u>20</u>, p. 469-489.

⁸⁶ NIST SRM 1867 and 1867a contain samples of tremolite, actinolite, and anthophyllite and are referred as "Uncommon Commercial Asbestos".

⁸⁷ The words in brackets are from the NIST SRM 1867a certificate.

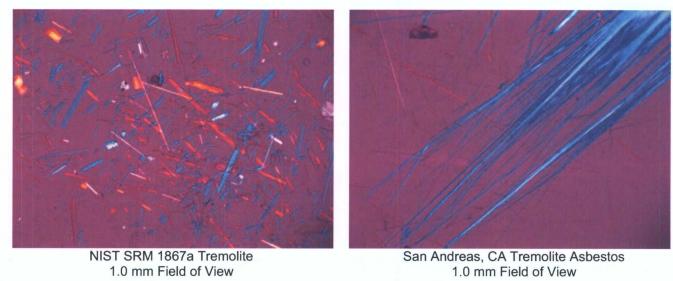


Figure 14. Tremolite from the NIST SRM 1867a sample (left) and a sample collected in San Andreas, CA (right) demonstrating the difference in appearance of the two varieties of Tremolite.

NIST SRM 1867a tremolite was examined to determine the extinction angle for those particles that are clearly asbestos and those that are clearly not asbestos. The asbestos fibers had extinction angles less than 10°, while the nonasbestos particles ranged up to 22°. Figure 15 illustrates the differences in extinction angle between these two different types of particles.

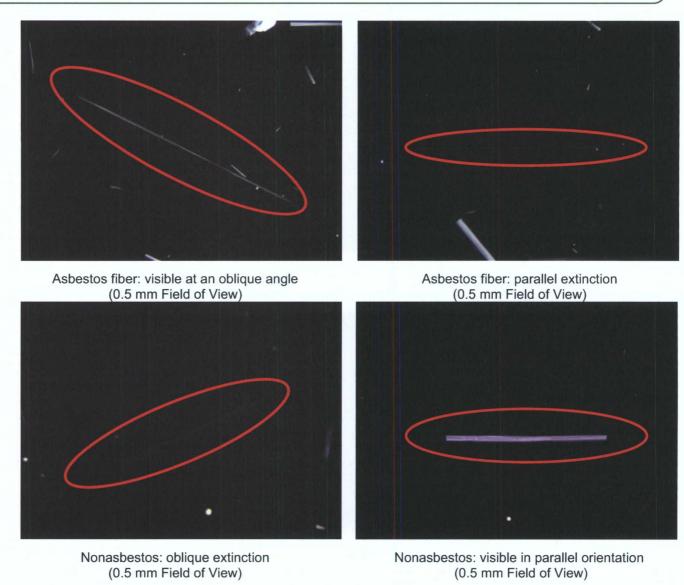


Figure 15. Photographs showing the extinction angles of asbestos and nonasbestos particles observed in NIST SRM 1867a.

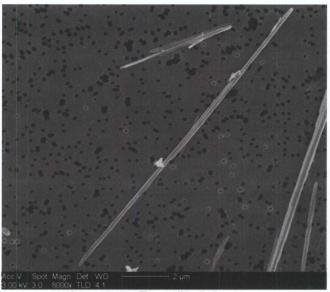
7.9 Fiber Terminations

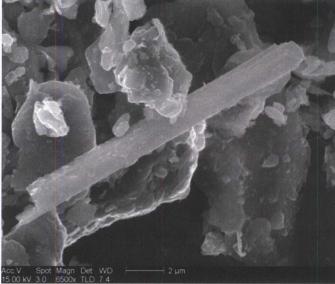
[EPA Request] During transmission Electron Microscopy ("TEM) or PLM, whether the R.J. Lee Group [sic] in the R.J. Lee Report would consider rounded terminations to be a definitive indicator that amphibole fiber is not an asbestos fiber.

Most fibers, at a sufficient magnification to observe the ends, have squared terminations. RJLG would not classify a particle with "rounded terminations" as nonasbestos solely on the basis of the terminations. If the particle exhibited the characteristics of asbestos 1) mean aspect ratios ranging from 20:1 to 100:1 or higher for fibers longer than 5 μ m; 2) very thin fibrils, usually less than 0.5 μ m in width; 3) parallel fibers occurring in bundles; 4) fiber bundles displaying splayed ends; 5) fibers in the form of thin needles; 6) matted masses of individual fibers; and/or 7) fibers showing curvature, RJLG would classify the

particle as asbestos. RJLG would not generally only evaluate the conditions of the particle terminations to determine whether it was asbestos or nonasbestos.

Figure 16 illustrates the particle morphology of tremolite asbestos fibers from Jamestown, CA and El Dorado Hills soil.





Jamestown Tremolite Asbestos

El Dorado Amphibole Particle

Figure 16. Comparison of the morphology of particles observed in the Jamestown tremolite asbestos sample with that of amphibole particles observed in the El Dorado Hills soil samples.

8.0 Verified Analyses

[EPA Request] Please provide a step-by-step description of the method and all the information sources used to perform the verified count in the R.J. Lee [sic] Report for its allocation of true and false positive values and provide supporting documents.

Verified analysis is the comparison of TEM data generated from at least two independent analyses of the same grid opening(s), using the same counting methodology. By comparing the following characteristics of the counted particles, it is possible to determine if the same particle was observed in the independent analyses of the grid opening: 1) relative location within a grid opening; 2) appearance; 3) orientation of the particle in the grid; 4) size of the particle; 5) particle morphology; 6) EDS and/or SAED information for the particle; and 7) particle identification by the analysts. Procedures describing these evaluations have been written by personnel from the National Institute of Standards and Technology.^{88,89,90}

⁸⁸ S. Turner and E. B. Steel (1994). Airborne Asbestos Method: Standard Test Method for Verified Analysis of Asbestos by Transmission Electron Microscopy – Version 2.0, NISTIR 5351.

In performing the evaluation of the Lab/Cor data from repeat analyses of the same grid openings, the count sheets for the original and QC analysis were located from within the produced materials. Both count sheets were converted to spreadsheets. After data entry and verification that the data entry was correct, the QC data was matched to the original data in the following steps:

Data from the same grid openings were placed side-by-side.

Data from within each grid opening were placed side-by-side on the basis of mineral identification (chrysotile or amphibole).

Within a mineral type, the data were arranged by particle morphology (as either individual fibers or bundles, or as part of a matrix or cluster). Where multiple matrices or clusters were found within a grid opening and mineral type, the types of substructures and size of the substructures were used to place the particles side-by-side within the spreadsheet.

Matching data were identified and enumerated. The count was based on the number of matched primary structures.

The following example is given for sample SRA-R04-100104. Only the data from the grid opening identified as C10 are included in this illustration.

After the data were placed side-by-side, as shown in Table 2, the particle dimensions were compared. Both analyses report a diffuse matrix that contains one fiber longer than 5 μm (MD1-1). While there is some variation in the dimensions of the fiber (7.5 μm vs 6.5 μm), the overall descriptions of this particle are similar enough to consider this as the same particle. However, the original analysis reported an additional fiber (F, 2.4 μm x 0.6 μm) that was not reported in the QA analysis.

Table 2. Alignment of data from two analyses of the same grid openings.

		Original A	Analysis		QA Analysis					
Loc.	Class	Length	Width	Aspect Ratio	Class	Length	Width	Aspect Ratio		
C10	MD1-1	7.5	4	1.9	MD1-1	7.8	3.8	2.1		
C10	MF	7.5	0.7	11	MF	6.5	0.7	9.3		
C10	F	2.4	0.6	4		No Structu	re Reported			

It is not possible to determine where the error occurred in the fiber count from this grid opening – was the fiber actually present but not verified or was it an error by the original

⁸⁹ E. B. Steel and J. A. Small (1985). Accuracy of Transmission Electron Microscopy for the Analysis of asbestos in Ambient Environments, *Anal. Chem.*, <u>57</u>, p. 209-213.

⁹⁰ S. Turner and E. B. Small (1991). Accuracy of Transmission Electron Microscopy Analysis of Asbestos on Filters: Interlaboratory Study, Anal. Chem., 63, p. 868-872.

analyst in reporting a structure that does not exist? However, one can conclude that there is a discrepancy with the fiber count on this grid opening.

The remainder of the grid openings were examined in a similar manner. Non-matching particle counts in the original analyses were highlighted as shown in the following Figure 17.

Original Analysis								QA Analysis						
Gr	No	Loc.	Prim	Tot	Class	Len	Wid	Asp	Prim	Tot	Class	Len	Wid	Asp
Α	1	A12	1	1	F	3.8	0.3	13	6	6	F	3.8	0.38	10
Α	2		2	2	F	3.5	1	3.5	No Structure Reported					
Α	3	B10	3	3	F	7.5	1	7.5	1	1	F	7.5	1	7.5
Α	3	B10	4		MD1-0	4	1.8	2.2	2		MD1-0	4.3	1.5	2.9
Α		B10		4	MF	4	0.45	8.9		2	MF	3.5	0.22	16
Α	3	B10	5	5		4.5	1.2	3.7	3	3	F	4	1	4
Α	4	A30	No Structure Reported						No Structure Reported					
Α	5	D2	6	6	F	7.5	1.8	4.2						
Α	5								7	7	F	2	0.6	3.3
Α	6	D31	7	7	F	2.5	0.7	3.6	9	9	F	2.5	0.4	6.2
Α	7	D23	8	8	F	4	1.1	3.6	8	8	F	3	0.25	12
Α		C10	9		MD1-1	7.5	4	1.9	4		MD1-1	7.8	3.8	2.1
Α	8	C10	2 17	9	MF	7.5	0.7	11		4	MF	6.5	0.7	9.3
Α	8	C10	10	10	F	2.4	0.6	4	No Structure Reported					
Α	9	C41	11	11	F	5	1	5	5	5	F	4.5	1	4.5
Α	10	C22	No Structure Reported						No Structure Reported					
Α	11	C14	No Structure Reported						No Structure Reported					
В	12	A30	No Structure Reported						No Structure Reported					
В	13	A11	No Structure Reported						No Structure Reported					
В	14	D21	No Structure Reported						11	11	F	5.8	1	5.8
В	15	C40	12	12	F	3.7	0.5	7.4	10	10	F	3.8	0.45	8.4
В	16	C11	No Structure Reported						No Structure Reported					
В	17	C24	13	13	F	9	0.8	11	No Structure Reported					
В	18	B1	No Structure Reported						No Structure Reported					
В	19	B20	No Structure Reported						Grid Opening Not Analyzed					

Figure 17. Illustration of the alignment of the particles from two analyses of the same grid openings of a sample showing the correlation of particle counts.

In the above example, no particle was observed in grid openings B22, C10, and C24 during the QC analysis. A second fiber was identified in grid opening D2 that was substantially shorter and thinner than the original analysis; the original fiber was not observed during the QC analysis. There is some question as to whether the particle in grid D23 is the same in each analysis primarily due to the difference in particle width. The problems identified in Figure 16 are found throughout the QA count sheets.

9.0 Reference Minerals

[EPA Request] Please provide all of the spectral data and supporting references for the R.J. Lee Group's [sic] mineral identifications relevant to the R.J. Lee Report, including all documentation of raw data, calculations, equations, and the supporting references.

RJLG utilized a number of reference standards, including rock specimens collected at El Dorado Hills. The standards also include the powder diffraction files published by the JCPDS, reference minerals NIST SRM 1866 and 1867, and mineral samples sold by Ward Scientific.

10.0 Optical Properties of Asbestos

[EPA Request] Please provide a copy of any written procedures employed by the R.J. Lee Group [sic] that describe how reference standards are used to verify the accuracy of an analyst's ability to correctly determine the optical properties of asbestos.

The procedures used to evaluate the accuracy of an analyst to determine the refractive indices of mineral fibers are contained in the laboratory QA/QC procedures. RJLG utilizes the NVLAP round robin tests and the New York ELAP tests to document the accuracy of this analysis.

11.0 Air Sample Analysis Procedures

[EPA Request] Please provide any written procedures or instructions given to analysts when the R.J. Lee Group [sic] performs a National Institute of Occupational Safety and Health (NIOSH) 7400 method analysis, an Asbestos Hazard Emergency Response Act (AHERA) method analysis or a California Air Resources Board AHERA- modified method analysis, including procedures regarding which aspect ratios are included in the count, whether or not all chrysotile or amphibole particles with the ratios that meet the method definition are included in the count, and any modifications to these methods. Please provide supporting documents, including any laboratory analysis bench sheets and reports with laboratory-identifying information redacted.

RJLG did not analyze any air samples during its review of the Lab/Cor data. However, if we had, the analyses would comply with the written procedures.

Given the concern expressed by EPA⁹¹ that RJLG arbitrarily modifies procedures or otherwise ignores certain particles of interest during its analyses, RJLG would welcome EPA observers to its laboratory to monitor the air sample preparation and analysis if the grids or filters analyzed by Lab/Cor are made available by EPA.

If El Dorado air samples are provided by the EPA, RJLG will analyze the air samples using the NIOSH 7400⁹² method supplemented by the NIOSH 7402⁹³ method using the TEM to apportion fibers into asbestos and nonasbestos fractions. Differentiating asbestos from nonasbestos particles is essential in mixed mineral environments where cleavage fragments or other nonasbestos particles may result in an increased PCM count. Using the

⁹¹ Sacramento Bee, April 2, 2006.

⁹² NIOSH Manual of Analytical Methods, Asbestos and Other Fibers by PCM, Method 7400, Issue 2, August 15, 1994, http://www.cdc.gov/niosh/nmam/pdfs/7400.pdf.

⁹³ NIOSH Manual of Analytical Methods, Asbestos by TEM, Method 7402, August 15, 1994, http://www.cdc.gov/niosh/nmam/pdfs/7402.pdf.

TEM (NIOSH 7402 method) particles with a minimum 3:1 aspect ratio will be analyzed, and energy dispersive x-ray spectra (EDS) and selected area electron diffraction (SAED) patterns will be recorded so that counted particles can be identified as asbestos or nonasbestos using the procedure approved by EPA Region 2.94

RJLG would supplement the NIOSH 7402 method by recording photomicrographs and SAED patterns additional to that required by the NIOSH 7402 method. Since the IRIS risk assessment procedures are based on the counting of asbestos, as defined in the EPA's El Dorado List of Terminology, 95 RJLG would use the criteria in Yamate, 96 OSHA ID 19133, and in the EPA bulk PLM methodology, 97 and supplement the analysis with SEM photomicrographs to document the morphology of individual particles. This is important because the NIOSH 7402 method was designed for measuring asbestos in a commercial environment, not for measurement of asbestos in mixed mineral dust environments.

EPA and ATSDR have indicated their intention to perform the risk assessment using the IRIS Model. This risk model requires the estimate of exposures to asbestiform fibers (not nonasbestos particles) be estimated. The fiber populations underlying IRIS resulted from commercial asbestos products being manufactured or used. In other circumstances where interferences would affect the reliability of the PCM measurement, IRIS requires the use of the NIOSH 7402 which classifies optically visible phase contrast microscopy equivalent (PCMe) fibers (particles with a minimum 3:1 aspect ratio and at least 5 μ m long that are visible in the TEM at 1000X magnification) as asbestos or non-asbestos. The proportion that is asbestos is multipled by the PCM concentration to derive the reported asbestos concentration. There is no provision for the use of TEM derived PCMe concentrations in IRIS.

Figure 18 illustrates the difference between asbestiform particles and particles from El Dorado Hills (mixed mineral dust environment).

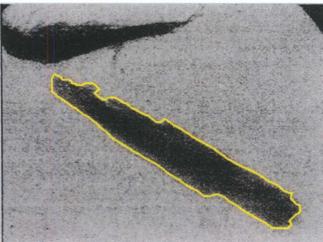
⁹⁴ P. Lioy, et al (2001). "Quality Assurance Project Plan: Assessment of Population Exposure and Risks to Emissions of Protocol Structures and Other Biologically Relevant Structures from the Southdown Quarry", January 24, 2001.

⁹⁵ Ecology & Environment (2004). El Dorado Hills Naturally Occurring Asbestos Multimedia Exposure Assessment El Dorado Hills, California, Quality Assurance Project Plan, Working Draft, EPA Contract No.: 68-W-01-012.

⁹⁶ G. Yamate, S. C. Agarwal, R. D. Gibbons (1984). "Methodology for the Measurement of Airborne Asbestos by Electron Microscopy", IIT Research Institute, Contract No. 68-02-3266, July 1984.

⁹⁷ R. L. Perkins and B. W. Harvey (1993). "Method for the Determination of Asbestos in Bulk Building Materials", U.S. Environmental Protection Agency, EPA/600/R-93/116, July 1993.



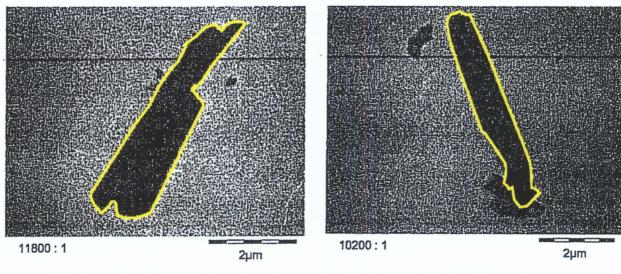


California Amphibole Asbestos

Lab/Cor Image of Counted Particle from El Dorado Hills Soil

Figure 18. Comparison of asbestiform particles with nonasbestos particles observed in the El Dorado Hills soil samples illustrating the morphological differences. The particles that Lab/Cor counted included particles with non-parallel sides which are outside the analytical method.

EPA Region 9 has suggested (Meer¹) that the definition of asbestos fibers within established asbestos analysis methods was not of particular interest – that any mineral particle that was at least 3 times longer than it was wide would be considered to be "asbestos." It is clear from our review of the data provided that Lab/Cor, EPA's contract laboratory, counted the particles in this manner and broadened the definition of asbestos fiber specified in the ISO 10312 method from particles which had substantially parallel sides to any particle having a minimum 3:1 aspect ratio, see Figure 19.



Sample NRA-R05-101004, #1 Actinolite, 5.2 µm x 1.2 µm

Sample SRA-R03-100704, #4 Actinolite, 4.8 µm x 0.65 µm

Figure 19. Exemplars of particles observed by Lab/Cor during the analysis of EDH air filters. Highlighted particle boundaries illustrate that Lab/Cor did not follow ISO 10312 counting rules.

It has been recognized by ATSDR, 98 NIOSH, 9 and Berman-Crump 8 that long, thin fibers have the most potential medical significance. RJLG would therefore additionally supplement the NIOSH methods by providing separate tabulations of countable structures into different size categories (i.e., longer than 10 μ m and less than 0.4 μ m in width, regardless of whether they are asbestiform or nonasbestos in nature; and longer than 40 μ m and less than 0.4 μ m, as have been suggested by Berman et al, 99 and more recently NIOSH, 9 as the principal components of an airborne fiber population correlating with disease). (Note that this is different than suggesting that only fibers longer than 40 μ m in the lung are relevant, rather, that airborne particle populations with measurable quantities of very long, thin fibers drive the statistical relationship with disease.)

RJLG will make all grids it analyzes available for review by EPA, either at RJLG or at EPA's designated laboratories. The latter recognizes that transfer of grids to another laboratory may result in damage to the grid and potential loss of particles.

Section 11 which follows is provided in anticipation of RJLG receiving air filters analyzed by Lab/Cor as has been requested.

⁹⁸ B. Case, M. Lippman, J. Lockey, E. McConnell, B. Mossman, G. Oberdorster, and W. Wallace (2002). "Expert Panel on Health Effects of Asbestos and Synthetic Vitreous Fibers (SVF): The Influence of Fiber Length; Premeeting Comments", ATSDR Agency for Toxic Substances and Disease Registry, October 29-30, 2002.

⁹⁹ D. W. Berman, K. S. Crump, Eric J. Chatfield, John M.G. Davis, and Alan D. Jones (1995). "The Sizes, Shapes, and Mineralogy of Asbestos Structures that Induce Lung Tumors or Mesothelioma in AF/HAN Rats Following Inhalation," Risk Analysis, 15, p. 181-195.

11.1 Laboratory Count Sheets

As observed by Mr. Smith in the EPA's 2004 audit of RJLG Monroeville Laboratory (Attachment C-11.1), RJLG has not maintained handwritten count sheets for a number of years. RJLG instead uses electronic recording at point-of-entry to document countable structures and produce reports. When EPA provides air filters, RJLG will, of course, maintain all data recorded electronically and provide copies.

11.2 Laboratory Notes

Any notes made by analysts during the course of analysis of air filters are electronically recorded and can be made available.

11.3 Logbook Pages

RJLG maintains an electronic logbook and will make the records available.

11.4 Sketches

RJLG does not normally maintain sketches of the particles analyzed but can do so in special circumstances. Since RJLG expects that the TEM samples prepared from the EPA El Dorado air filters will be transferred from the TEM to the SEM for additional imaging, RJLG will maintain and make available copies of any sketches recorded.

RJLG requests that any sketches recorded by Lab/Cor, but which have not been produced in response to previous requests, be made available for review.

11.5 Images

All images recorded are saved electronically. RJLG will make copies of any images recorded available.

RJLG requests that copies of images recorded by Lab/Cor, but which have not been produced in response to previous requests, be made available for review.

11.6 Spectra

All EDS spectra are electronically recorded and maintained. RJLG will provide copies of all recorded spectra.

11.7 Diffraction Patterns

Selected area electron diffraction (SAED) patterns are recorded electronically and processed to determine orientation relative to the electron beam, major crystal faces and potential mineral species of particles. These data are stored electronically. RJLG will make copies of SAED data available.

More than 30% of the SAED patterns provided by Lab/Cor were illegible scans of printed documents. It is apparent that Lab/Cor maintains electronic copies of most, if not all,

SAED patterns. Given EPA's position¹⁰⁰ that RJLG failed to obtain all relevant information and samples before commenting on the laboratory analysis, RJLG requests that all such electronic copies be produced for review.

11.8 Chain of Custody Forms

RJLG will maintain and provide copies of all chain of custody documentation.

11.9 Sample Tracking Sheets

RJLG maintains an electronic tracking system and will make the records available.

11.10 Quality Control ("QC") Sample Information

RJLG will maintain and provide information for QC samples analyzed.

11.11 Required Calibrations

RJLG maintains all required calibration records as part of its laboratory certification requirements for the NVLAP, AIHA, NELAC, and CA ELAP certification processes and will provide calibrations relevant to the time frame in which EPA air samples are analyzed for the instrumentation used in processing the samples.

Lab/Cor's data production did not include any relevant calibration data, including camera constants for SAED analysis, reference asbestos mineral photographs or EDS patterns used to calibrate their EDS system(s). During its review of Lab/Cor's data, RJLG went to extraordinary lengths to determine such information given the data provided. Given EPA's concern¹⁰¹ that RJLG's analysis was lacking or limited by the available information or samples, RJLG renews its request that all such data and information be produced by Lab/Cor.

12.0 Bundles

[EPA Request] How would the R.J. Lee Group,[sic] when using an asbestos regulatory air analytical method (i.e., NIOSH or AHERA) or International Standards Organization ("ISO") method 10312, count bundles of asbestos fibers?

The referenced analytical methods provide definitions of bundles and instructions as to how these asbestos structures are to be counted. RJLG complies with these instructions.

EPA Region 9 suggests³ (2006, page 7) that RJLG ignored the presence of bundles in the Lab/Cor data when performing its evaluation of fiber dimensions. Our experience with data from other laboratories indicates that some record the width of the component fiber while others record the overall width of the bundle. It was not clear what Lab/Cor's procedure was in recording the dimensions of bundles. Therefore, bundles were not

 $^{^{100}\} EPA$ April 20 2006 Response, page 3.

¹⁰¹ EPA 2006 Response, page 3.

included in the analysis of fiber dimensions, but they were included in any concentration calculation.

The available data suggest the "bundles" identified by Lab/Cor may not even be bundles but that they may be cleavage fragments with evidence of striations. Fewer than 5% of all amphibole structures counted were bundles, hardly indicative of an asbestos fiber population. Eighty-five percent (85%) of the "bundles" were wider than 0.5 μ m and 60% were wider than 1 μ m (they basically had the same dimensions as the "fibers"). There were no photographs of bundles produced by EPA and very few diffraction patterns. RJLG requests that Lab/Cor's TEM grid preparations with reported bundles be produced so that the actual nature of these particles can be determined.

13.0 Selected Area Electron Diffraction Patterns

[EPA Request] How the R.J. Lee Group, [sic] when analyzing the selected area electron diffraction ("SAED) pattern of an amphibole mineral fiber, would distinguish between asbestos and non-asbestos mineral fibers?

It is recognized that amphibole asbestos fibers preferentially orient along the {100} crystal face. (The symbol {hkl} represents the Miller indices of all symmetrically equivalent faces.) The principal cleavage direction in amphiboles is parallel to {110}. An amphibole crystal that grew as a fiber should show {100} faces but lack {110} faces. An amphibole crystal that is a cleavage fragment will show {110} faces and may or may not show {100} faces. Indexing the faces of a crystal and documenting a {110} face will help to identify the crystal as a cleavage fragment.

SAEDpatterns are measured to determine the angles and distances (d-spacings) between atomic planes [hkl] in a crystal. This information is used for mineral identification by comparison of these results with standard reference patterns. Once a pattern is indexed, the crystal zone axis [uvw] parallel to the direction of the incident electron beam can be determined.

The relationships between the zone axis, angular tilt, and crystal faces are illustrated in Figure 20. An example of measured crystal faces are shown in Figure 21 and Figure 22.

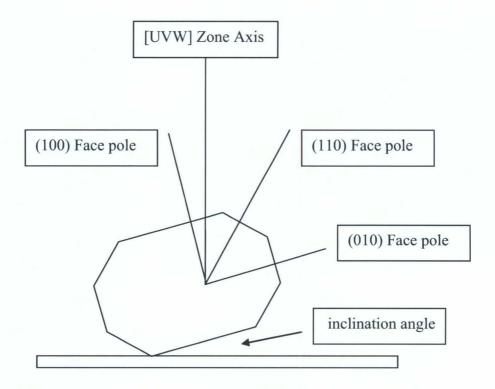


Figure 20. Diagram showing relationship between TEM zone axis and crystal faces

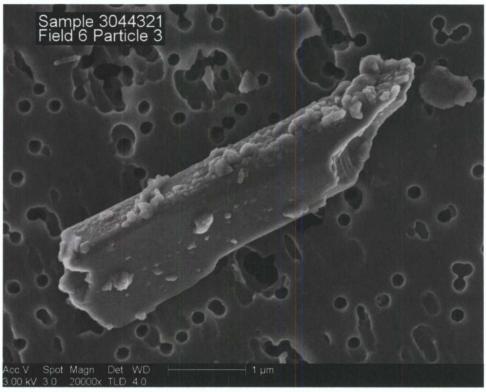


Figure 21. SEM Micrograph of an amphibole crystal from an El Dorado Hills soil sample

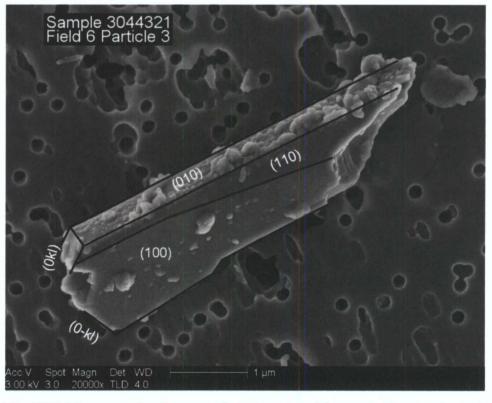


Figure 22. SEM Micrograph of an amphibole crystal from an El Dorado soil sample

with superimposed crystal faces.

The crystallographic zone axes from the Lab/Cor data are plotted in stereographic projection in Figure 23. Since the same zone axes were found multiple times, the data in each figure are contoured to show the concentration of the zone axes. The average zone (vector mean of the data) plots essentially along the (110) face pole. The majority of the zone axes (~70%) lie in the region between the (110) face pole and the [110] zone axis. This is indicative that the majority of the crystals analyzed are not asbestos fibers. Figure 24 shows the results of RJLG's analysis of El Dorado soil samples which indicates a population of nonasbestos particles. For comparison, Figure 25 shows the clustering of the zones for tremolite asbestos from Jamestown, CA (a tremolite used in animal studies⁷). The zone axes are clustered around the (100) face poles with a few near (1-10), indicating these are from a population of asbestos fibers. Note: Particles that exhibit SAED patterns near (100) are not, in and of themselves, indicative of an asbestos fiber, but they do present evidence that the fiber may be asbestos. Additional information, such as particle morphology and/or chemistry, is needed for a final determination.

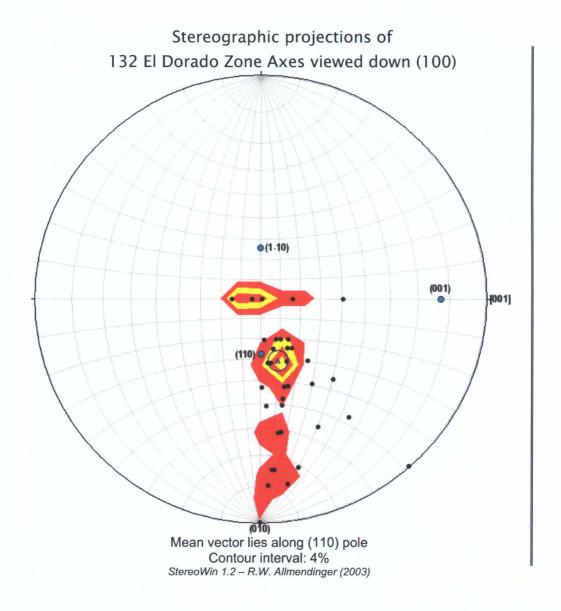


Figure 23. Stereographic projections of 132 identified zone axis patterns from the Lab/Cor data set. The graph is indicative of a population of nonasbestos particles.

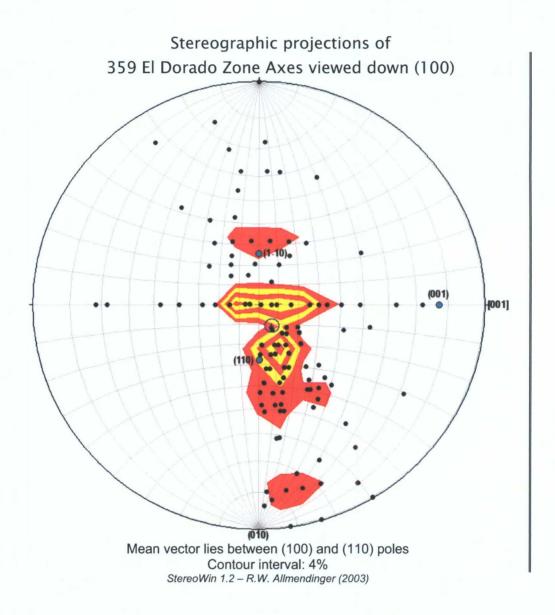


Figure 24. Stereographic projections of 359 identified zone axis patterns (RJLG's analysis of El Dorado Soil samples). The graph is indicative of a population of nonasbestos particles.

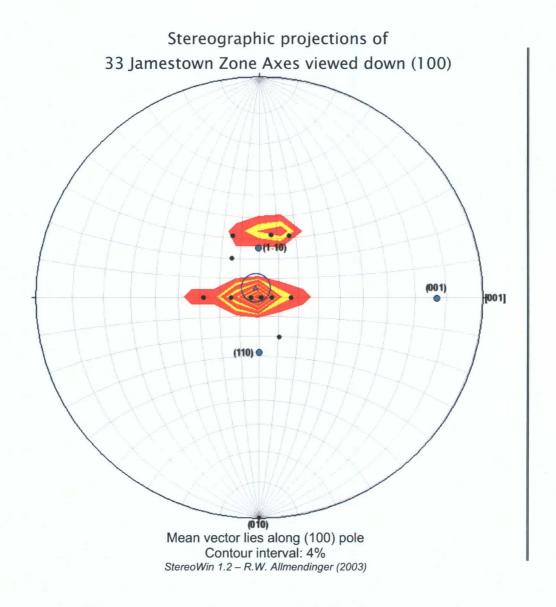


Figure 25. Stereographic projection of the zone axes from a population of tremolite asbestos from Jamestown, CA. The zones are centered around the (100) pole.

14.0 Rock Samples

The rock samples collected in and around the El Dorado Hills test site on January 10, 2006, were analyzed using PLM in general accordance with the principals of geology. Verification of the mineral identification was performed using PLM¹0². Minerals that could potentially be amphibole were removed from the rocks, lightly ground in a mortar and pestle, and deposited onto SEM stubs. The particles on the stubs were examined in an SEM to obtain images of representative particles. In addition, finely ground particles were deposited onto a filter and examined in the TEM for elongated particles. EDS spectra of representative particles that were at least 3:1 aspect ratio were collected.

14.1 Documents, Count Sheets, EDS spectra

Documents, count sheets, EDS spectra, and photographs generated during the analyses of the rock samples are provided in Attachment C-14.1 to this response.

14.2 Images

All photographs related to the rock samples are provided in the attachment described in Section 14.1 above. These include field photographs taken at EDH as well as PLM, SEM, and TEM photographs of representative particles.

14.3 Chain-of-Custody

The chain-of-custody for the rock samples is provided in Attachment C-14.3.

15.0 Elutriator Tests

Four soil samples were evaluated using an elutriator procedure described in "Modified Elutriator Method for the Determination of Asbestos in Soils and Bulk Materials," by Berman and Kolk.⁴⁷ Generated filters from the elutriator were analyzed in general accordance with ISO 10312.

15.1 Counts Sheets and Laboratory Notes

The letter report, TEM count sheets, images, EDS and SAED patterns for the elutriator test sample analyses are provided in Attachment C-15.1. Laboratory notes related to the generation of the air filter for each sample are included.

15.2 Chain-of-Custody

The chain-of-custody for the soil samples is provided in Attachment C-3.5a.

^{102 40} CFR Part 763, Appendix E to Subpart E.

16.0 Qualifications of Richard J. Lee, Ph.D.

Richard J. Lee, Ph.D., has been involved in the development of methods for the identification of asbestos and other airborne particles for more than 30 years. Dr. Lee authored the first computer software for the analysis of electron diffraction patterns and the automated sizing and chemical analysis of asbestos and other particles. Dr Lee was an active participant in the first ASTM committee whose goal was to develop and test a TEM method for the analysis of asbestos, and was a co-author of the publication that resulted.

Dr Lee has served, from time to time, as an advisor to EPA for more than 25 years, beginning with the design of an EPA laboratory for the analysis of asbestos and other particulate, and serving as a peer reviewer/consulting expert in the development and writing of EPA's Yamate method. Dr. Lee authored the TEM analysis method instituted as part of the AHERA rules. Dr. Lee has performed the laboratory analysis underlying more than 30 EPA projects, including EPA's assessment of the airborne asbestos concentrations in public buildings, and has served as a peer reviewer on other EPA projects. Dr. Lee served on the HEI peer review panel to assess the significance of asbestos in public buildings, and was one of the authors of the landmark report that resulted from that review.

Dr. Lee and his staff actively consulted with and supported EPA Region 2 in evaluating contamination in NYC buildings that were impacted by the Events of 9/11. Dr. Lee and his staff designed, implemented, provided oversight and conducted sampling and laboratory analyses for building remediation. His group also planned, organized, developed protocols and conducted health and safety and community air quality monitoring programs in and around the impacted deconstruction/demolition of one of the buildings. Dr. Lee and his staff reported project status and results to EPA Region 2 on a weekly basis during the course of the multi-year study. Dr. Lee and his staff actively supported EPA's Office of Research and Development in the development of a method to evaluate dusts from the Lower Manhattan district associated with the World Trade Center disaster. Dr. Lee and his staff supported EPA's investigation of potential emissions of tremolite asbestos from the Southdown Quarry in New Jersey. Dr. Lee has served on ASTM committees to define methods for the analysis of asbestos.

Dr. Lee has served as an expert witness in state and federal courts in numerous asbestos related cases over the last twenty years. Dr Lee is currently retained by W. R. Grace & Co. in the matter of United States of America v. W. R. Grace, et al (CR-05-070M-DWM D. Montana). Dr. Lee has published the most extensive surveys of asbestos concentrations in public buildings ever performed. Dr. Lee has also published over 150 papers.

Dr. Lee supervised the data review and the laboratory analysis conducted in conjunction with RJLG's review of EPA's analytical data from the El Dorado Hills Asbestos Evaluation Project.

Response of RJ Lee Group to EPA Region 9 (Meer) Letter dated March 9, 2006

Dr. Lee holds a Ph. D. in theoretical physics, and was previously employed by US Steel Research where he was active in the development of asbestos analysis methods before forming RJ Lee Group in 1985.